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RSIC-437

FUNDAMENTALS OF SOLID STATE WELDING AND THEIR APPLICATION TO BERYLLIUM, ALUMINUM, AND STAINLESS STEEL

by
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and
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Redstone Scientific Information Center

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REDSTONE ARSENAL, ALABAMA

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Columbus, Ohio

Contract No. DA-01-021-AMC-11706(Z)

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ABSTRACT

This report presents the results of a state-of-the-art survey of solid state diffusion welding and roll welding of aerospace metals. Particular emphasis is given to beryllium, high-strength aluminum alloys, and Type 321 stainless steel.

The report is divided into four major categories:

1) A general survey of solid state diffusion welding, including a description of a real surface, factors influencing microscopic plastic flow, diffusion, grain growth, dissimilar metal welding, and use of intermediate materials.

2) A description of solid state welding methods, including roll welding, roll-welding theory, advantages and limitations of welding methods, and joint designs that are suitable for diffusion welding and roll welding.

3) Nondestructive testing techniques for solid state welded joints and their applicability.

4) A survey of similar and dissimilar metal diffusion welding the selected metals.

On the basis of the information obtained, general areas in which future research is desirable are presented.

FOREWORD

The purpose of this report is to review available information concerning diffusion welding and roll welding of aerospace alloys. Primary emphasis is given to beryllium, high-strength aluminum alloys, and Type 321 stainless steel. Briefly, the areas included in the present report are:

1) Effect of process variables on weld properties.

2) Metallurgical characteristics of the welds produced.

3) Processing techniques and present limitations.

4) Nondestructive techniques for inspection of weld integrity.

This survey was requested by the NASA Manufacturing Engineering Laboratory at NASA's George C. Marshall Space Flight Center, Huntsville, Alabama.

Information for this report was obtained from the literature cited. Assistance was given by the Redstone Scientific Information Center.

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Section I. INTRODUCTION

The fabrication of structures for aerospace applications places strict requirements on the methods used for joining. These requirements encompass such factors as minimum weight, adequate properties, ease of joining, and reliability. Possible joining methods include fusion welding, brazing, mechanical fastening, adhesive bonding, and solid state welding. It is with the last, solid state welding, that this report deals.

Solid state welding is any process in which two or more solid phases are metallurgically joined without the creation of a liquid phase. The term metallurgically joined is used to describe weld formation by the action of atomic forces rather than solely by mechanical interlocking or by an adhesive.

1. Definitions

Solid state joining includes a number of welding processes. These processes will be referred to as this report progresses. It is, therefore, appropriate to define at least a part of the terms which will be encountered.

a. Gas Pressure Bonding

Gas pressure bonding is a diffusion welding process which uses a pressurized gas to impart the welding pressure. This process is described in more detail later in this report.

b. Solid Phase Welding

Solid phase welding is the same as solid state welding. Joining occurs entirely in the solid state with or without measurable deformation.

c. Self Welding

Self welding is the joining of parts of the same metal, regardless of the joining method used.

d. Recrystallization Welding

Recrystallization welding is a weld near which recrystallization has occurred.

e. Press Bonding

Press bonding is any joining method in which joining pressure is applied by a press.

f. Vacuum Bonding

Vacuum bonding is joining performed in a vacuum.

g. Pressure Bonding

Pressure bonding is any process in which pressure is used to complete the weld.

h. Cold Welding

Cold welding is a pressure-welding process that uses pressure to initiate the weld.

i. Forge Welding

Forge welding includes several welding processes that use heating by a forge or furnace and some form of pressure application.

j. Roll Welding

Roll welding is a forge-welding process that produces a weld by heating the parts to be joined in a furnace and applying pressure with rolls.

k. Gas Welding

Gas welding produces a weld by heating the parts with a gas flame with or without pressure application and with or without a filler metal.

l. Induction Welding

Induction welding uses heat produced by the resistance of the metal to the flow of induced electric current, with or without pressure application.

m. Resistance Welding

Resistance welding uses pressure and heating that is produced by the resistance of the metal to the flow of electric current in a circuit of which the parts to be joined are a part.

n. Thermocompression Bonding

Thermocompression bonding is essentially the same as forge welding.

Solid state welding possesses a number of important advantages over other joining techniques which strongly recommend its use for fabrication of aerospace structures. The advantages will be illuminated as this report unfolds. As a joining method, solid state welding can be divided into two classes.

2. Diffusion Welding

In this type of welding, diffusion is the principal factor in the formation of a weld and deformation occurs only on a microscale. Because diffusion rates at room temperature are usually quite low, diffusion welding generally requires contact of relatively clean metallic surfaces at elevated temperatures. For convenience, the diffusion welding process can be considered as a two-stage process, even though the two stages may occur simultaneously.[1] The two stages are:

1) Microscopic plastic deformation resulting in intimate metal-to-metal contact.

2) Diffusion and grain growth to complete the weld and ultimately eliminate the interface formed in Stage 1.

Some authors consider that the interface must be entirely eliminated before a diffusion weld is complete; however, at some point prior to the elimination of the interface, the weld generally develops a strength as great as that of the base metal. Although these two definitions of a diffusion weld are related, a definition based on joint strength is probably preferred for engineering applications.

Diffusion welded structures possess the following advantages and properties:

1) Welds can be made that have essentially the same physical, chemical, and mechanical properties as the base metal.

2) Welds can be made with no impairment of the base material's properties.

3) Metals can be joined without the creation of porosity, erosion, or embrittlement.

4) A cast structure is not created in the weld.

5) Welds can be formed below the recrystallization temperature of nearly all metals.

6) Low remelt temperatures such as those observed after brazing can be avoided.

7) Weldability is largely independent of material thickness.

8) Continuous, leaktight welds can be formed.

9) Dissimilar metal couples can be joined.

10) Numerous welds can be made simultaneously.

11) Welding and heat treating of parts can be performed simultaneously.

12) Flexibility in joint design is possible.

While many similar or dissimilar metal couples may be joined by solid state diffusion welding, considerable emphasis has recently been placed on the reactive metals (Ti, Be, Zr) and the refractory metals (Cb, Ta, Mo, W). Advanced applications for which diffusion welding shows considerable promise for further development and usage are given elsewhere.[2,3,4,5]

3. Deformation Welding

Deformation welding includes those techniques in which gross plastic flow is the major factor in weld formation and diffusion is not necessarily required, although it may contribute to the formation of a weld if the deformation is carried out at high temperatures. Two theories exist that attempt to explain the mechanism of deformation welding. The "film theory" proposes that when two clean metallic surfaces are brought into contact, welding will occur. Plastic deformation, in this theory, is necessary to break up surface films that preclude intimate metallic contact and weld formation. Elastic recovery, which tends to

rupture welds upon removal of the welding pressure, may also be of considerable importance in deformation welding. The "energy barrier theory" proposes that, even if clean surfaces are brought into intimate contact, no weld will be formed. The theory states that an "energy barrier" must be overcome before welding can occur. Deformation is one form of energy that can surmount this barrier.

Deformation welded structures possess several advantages and properties that are similar to those given for diffusion welding. In addition, work hardening and strengthening can be accomplished during the welding process. For this strengthening, the welding temperature must be below the temperature at which annealing will remove strain hardening.

It can be observed that the process definitions lean heavily toward actual welding processes. The two basic classes of solid state welding (e.g., diffusion welding and deformation welding) are based on the mechanism of welding rather than any specific joining method. The major difference pointed out between diffusion welding and deformation welding is the amount of deformation used to join metal parts. In the former, the deformation that may occur is only that required to bring surfaces, having a roughness that is commonly encountered, into intimate contact. The deformation is further restricted to a narrow region (on a microscale) near the faying surfaces. In the latter type of welding, these restrictions do not apply. Diffusion is required in diffusion welding but may or may not occur during deformation welding, depending on the welding temperatures used.

Another point which should be made is that, technically, either the term "bonding" or "welding" may be used when describing any of the solid state joining processes, on the basis of definitions given by the American Welding Society, although the term "welding" is probably preferable. The term "Roll Bond" is a registered trademark, so the term "roll welding" is recommended for general use. Currently, the term "diffusion bonding" is probably the most commonly used term for what is called "diffusion welding" in this report; therefore, the terms "diffusion welding" and "deformation welding" will be used throughout this report. The term "roll welding" will be used for that particular deformation welding process.

Section II. GENERAL SURVEY OF SOLID STATE DIFFUSION WELDING

In addition to summarizing and collating a large number of reference articles, an attempt has been made in this section to analyze this information in such a manner as to permit the reader to gain an understanding of the mechanisms of diffusion welding.

As stated in the Introduction, deformation during diffusion welding occurs only on a microscale as opposing surfaces are brought into intimate contact. Diffusion is the principal factor in the formation of a weld. Although a straightforward, two stage welding model may be easily visualized, the several phenomena (mechanical, chemical, and metallurgical), which proceed during joining, become quite complex. The interactions of the factors that affect diffusion welding produce a situation that resists an unequivocal separation and analysis of those factors.

Several comprehensive literature surveys on solid state welding have been prepared.[6,7,8,9,10,11] An additional state-of-the-art summary is currently being prepared under the sponsorship of the Welding Research Council.

One recent literature survey[7] presented a number of conclusions which should aid the student of this subject in gaining a better understanding of diffusion welding. This survey was the initial step in a program devoted to the development of diffusion welding procedures suitable for refractory metal aerospace structures. A major goal was to survey and analyze concepts that may be employed to activate the diffusion welding process. Experimental studies subsequently examined these concepts in order to develop improved diffusion welding techniques. The following conclusions from the initial literature survey were made:

1) Solid state diffusion welding is a duplex process consisting of plastic deformation to provide intimate contact and oxide disruption, and creation of metallic bonds by diffusion and grain growth across the original interface. Barriers to diffusional phenomena must not be present at the interface.[12, 13]

2) Soft, metallic intermediate layers aid the attainment of surface conformance and can restrict deformation to the region between the parts to be joined. Elevated temperatures play an important role by lowering the yield strength of the intermediate.[2, 11, 12, 13, 14]

3) Constituents that have high diffusivities in the materials to be joined promote diffusion welding when placed at the interface prior to contact. If these elements diffuse by a grain boundary mechanism, they may introduce hot shortness, recrystallization, or gain growth.

4) Intermediate metals that move by a volume diffusion mechanism and that have high diffusivities promote diffusion welding. Some solubility with the base metal, formation of a high melting solid solution with the parent metals, and a small atomic diameter[15] are criteria for such a material.

5) Residual or localized cold work may aid the achievement of intimate surface contact by recrystallization near the interface. During a period of metallurgical instability such as recrystallization, abnormally high ductility is observed. The assistance by cold work and recrystallization on the second or diffusional stage in the welding process is questionable.[2,16,17]

6) Other sources of metallurgical instability may also promote diffusion welding in the way described above. Such phenomena might include allotropic transformations, eutectoid transformations, or states near to solubility limits. Lattice parameter changes or other metastable conditions promote temporary ductility.[17,18]

7) When the material yield stress is low, surface preparation is not critically important.[12,15] When diffusion is the primary mechanism of weld formation, surface preparation becomes significant.[2,11,13]

8) Barriers that inhibit diffusion and that are usually brittle, such as intermetallics or interstitial compounds, tend to prevent welding. Diffusion against the concentration gradient may occur.[2,11,12,14]

9) Interfacial melting must be avoided as well as the formation of low melting point eutectic phases.[2]

10) When welding temperatures are high enough to provide adequate ductility, surface roughness is not important.[2,11,13] Smooth surfaces are preferred when plastic deformation is restricted. The reason for this may be connected with the effects of surface energy or material prestrain.

The authors of the present report are not in full agreement with all of the conclusions given above, particulary conclusions 7) and 10). In general, however, these statements provide a brief summation of several important aspects of the diffusion welding process.

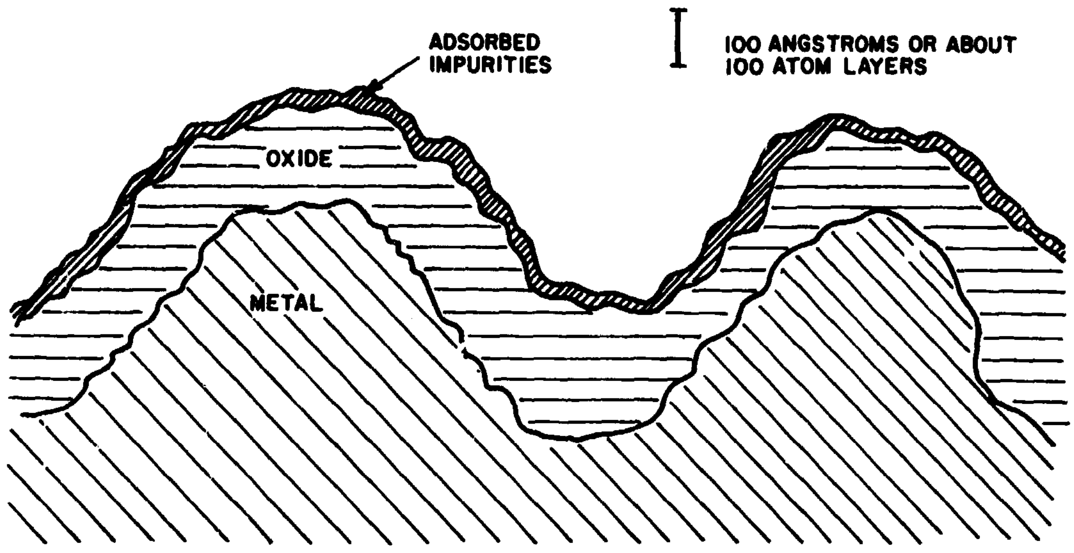


Figure 1. Schematic Diagram of a Real Surface

1. Description of a Real Surface

To adequately describe a real surface, there are two dominant characteristics, roughness and impurities, which must be considered. A schematic diagram of a real surface is shown in Figure 1. On a microscopic scale, surfaces are extremely rough.[10, 19] Mechanically prepared surfaces consist of grooves or scratches.[20] In addition to these first-order scratches, secondary ridges exist on the faces of the primary scratches, as observed with the electron microscope. Third and fourth order grooves may also be present, although their existence is believed to be unlikely. Both the primary and secondary forms of roughness have an obtuse included angle at the peaks of the asperities. Finely polished, abrasively prepared surfaces are topographically similar to rough surfaces, although the included apex angle of the scratches is believed to be more obtuse. The most highly polished surfaces, for example, will have peak-to-valley distances of roughly 500 Å.[21, 22] Ground surfaces will be many times rougher than this. The initial contact area produced when metallic surfaces are brought together will be very much less than the nominal surface area, possibly 10^{-6} times the nominal area.[23, 24] Because of surface roughness, a load, normal to the interface, will be supported by asperities that will plastically deform until the actual area of contact is capable of supporting the applied force. The true area of contact is proportional to the load and is independent of the size or shape of the surfaces. An applied tangential stress will

increase the area of contact for a given load.[19, 25] This will also assist the displacement of oxide layers and thereby promote bonding. Tangential shear stresses have been shown to promote instantaneous adhesion when coupled with compressive stresses.[26]

Furey[27], in a study of friction and lubrication, gave the values contained in Table I for average surface roughness in microinches (centerline average) of AISI 52100 steel cylinders prepared on a lathe.

Table I. Average Values for Surface Roughness

Mode of Surface Preparation	Average Roughness, μ in. (CLA) Parallel to Cylinder Axis	Around Cylinder Circumference
Sand blasting	88.3	87.3
Grinding or polishing with aluminum oxide abrasive cloth		
80 grit	40.5	19.6
120 grit	23.5	12.7
180 grit	22.2	12.5
240 grit	10.7	5.6
320 grit	6.2	5.2
500 grit	4.2	3.4
SiC polishing cloth	3.1	3.4

All metallic surfaces exposed to air are covered with an oxide film 10 to 10^5 A thick. The thickness of this film will vary with the chemical properties of the metal and the environment. The oxide film on noble metals may only be several atomic layers thick while, on most other metals, it is much thicker.[19] In air, at atmospheric pressure, a monolayer of gas will form on a surface in about 4×10^{-9} seconds while at a pressure of 10^{-6} torr, approximately 3 seconds are required.[28] Layers of adsorbed atoms and molecules will also be present on the oxide film.

2. Factors Influencing Microscopic Plastic Flow

As mentioned in the last section, when surfaces are brought into contact, they meet over only a very small area. This area is essentially a function of the shape and roughness of the two surfaces. The application of pressure will increase this area of contact until it is equal to or actually somewhat greater than the nominal surface area, measured macroscopically. Factors which must be considered are the applied load, the shape, the size, the number, and, in particular, the depth of the asperities and the yield pressure of the metal.[29,30] As the surfaces are brought together, the asperities deform to a larger radius of curvature, as shown in Figure 2, until the yield pressure of the opposing surface is exceeded. At this point, penetration by the asperities occurs, and the void areas are filled by displaced metal. This description is applicable to metals that both do or do not work harden. Penetration by the asperities will occur sooner for metals that work harden. When a hard metal and a soft metal are joined, little deformation of the harder metal will occur. As penetration proceeds, displaced metal forms new points of contact with smaller asperities in the voids and the true area of contact is further increased. If specimens are brought into contact at low temperatures and the load is released, residual stresses may be capable of fracturing the few welds formed. At higher temperatures, residual stresses will be removed by annealing, and welding will occur more readily. Frictional forces between the surfaces during penetration of the asperities can become significant, in effect, by increasing the yield pressure of the metals.

As the contacting surfaces become smoother, more area is in contact for a given load and the pressure is less than for the initial areas of contact of rough surfaces. The Vickers harness, measured with a square based, pyramid shaped indentor, is a measure of the applied load divided by the surface area of the indentation. The mean pressure is based on the projected area of the indentation and equals the Vickers hardness divided by 0.927. The hot hardness and surface roughness are sufficient parameters to estimate the pressure required to achieve a given fractional area of contact. Theoretical analysis suggests that the Vickers hot hardness is the pressure required to produce intimate contact while experiments showed the actual pressure to be less. This discrepancy arises because the theory does not account for the accumulation of displaced metal during indentation of one surface by asperities of the other. It was also observed that, as penetration of the asperities proceeded, interfering flow patterns arose and the accumulation of displaced metal occurred, as discussed earlier. Finally, a point was reached where very small amounts of penetration and, therefore, strain increased the contact area substantially. For pure copper, it was

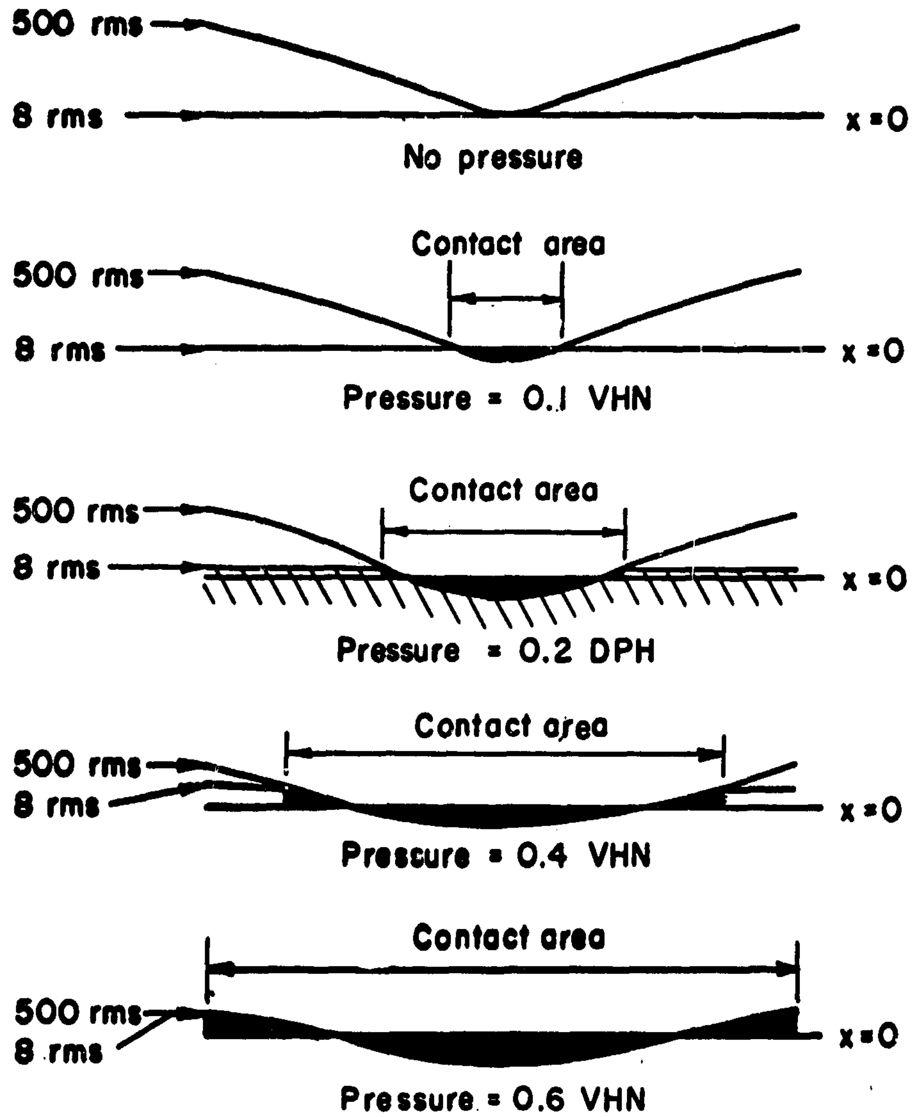


Figure 2. Representation of the Deformation Process [29]

shown that the contact pressure would have to be about 0.6 of the Vickers hot hardness for complete contact. This evaluation was made for a 500-μ inches rms shaped surface pressed into an 8-μ inches rms ground surface. It was also shown that the pressure required was inversely proportional to the surface roughness and directly proportional to the number of points of contact raised to the n-2/n power where n is the Meyer strain-hardening coefficient.

3. Diffusion

Because diffusion is the predominant factor in the creation of diffusion welded joints, some theory and review of pertinent articles are presented below. More detailed information may be found in the cited references. Although specific data are not presented in this report, values for the diffusivities in numerous systems may be found in References 6, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, and 42.

a. Fundamentals of Diffusion

Background information on diffusion phenomena is presented in this section. Basic equations that mathematically express these occurrences and atomic mechanisms, which are used to describe diffusion phenomena, are discussed.

(1) Diffusion Equations. Basic to an understanding of diffusional phenomena are Fick's laws.[31] The first law states that the flux of a component is proportional to the concentration gradient, for simplicity, in the x-direction only. This is expressed as $J = -D(\partial c/\partial x)$ where the flux, J, is the mass of atoms crossing a plane of unit area per unit time ($mass/L^2/t$). The concentration gradient, $\partial c/\partial x$, has units of $\frac{mass/L^3}{L}$ and D, the diffusion coefficient or diffusivity is expressed in terms of L^2/t. In these expressions, L is length and t is time. Fick's second law of diffusion may be expressed (in one direction) as $\partial c/\partial t = \partial/\partial x\,(D\,\partial c/\partial x)$, where the symbols have the same meaning as above. From these equations, solutions to fairly simple problems may be found. The following, applicable to diffusion welding situations, are good examples.

For a thin film of solute between the ends of long rods of solute free material, the concentration of solute along the bars is given as:

$$c = \frac{\alpha}{2\sqrt{\pi d t}} \exp\left(-\frac{x^2}{4Dt}\right)$$

where α is the quantity of solute, x is the distance normal to the thin film, D is the diffusivity, and t is the time.[31] Thus, after a certain time, the solute concentration at some distance from the original interface may be approximately calculated. The diffusivity, D, at the temperature involved and of the species in question, must be known. This solution assumes that D is constant. Solutions to a number of other diffusion problems are readily available. In real situations, D is not constant but varies with direction, composition, and temperature. Methods exist for incorporating the variable diffusivity.

If two metals that display mutual solid solubility are placed in contact, the concentration of one atomic species in the other as a function of distance and time may be expressed as:

$$c(x, t) = \frac{C_o}{2} \left[1 - \operatorname{erf} \left(\frac{x}{2\sqrt{Dt}} \right) \right].$$

C_o is the initial concentration and erf(z) is the Gaussian error integral, the values of which have been tabulated.[31, 45] Solutions for other problems are available, e. g., couples having partial solid solubility and growth of an intermediate phase[44].

(2) Atomic Mechanisms. Diffusion results from atomic motion, which occurs by discreet jumps from one lattice position to another. Three principal mechanisms, discussed below, are believed to be responsible for volume diffusion in crystalline solids. These mechanisms are shown schematically in Figures 3 and 4.

(a) Interstitial Mechanism - This type of diffusion occurs when an atom moves from one crystalline interstitial site to an adjacent interstitial site without radically disturbing the atoms at the lattice points, as seen in Figure 3. To accomplish this motion, some lattice expansion is required as the interstitial atom changes position. The energy required to overcome this barrier decreases as the size of interstitial atom decreases, relative to solvent atom size. Atoms of H, N, B, C, and O are interstitial in most metals. If the interstitial atom is large, this mechanism will not be operative and another mechanism must become active. Interstitial diffusion in substitutional alloys is not a likely occurrence. This is because a large amount of energy is required to "squeeze" substitutional atoms into interstitial positions.

(b) Vacancy Mechanism - In all crystals, vacant lattice sites exist. This mechanism consists of atoms moving to unoccupied lattice positions, shown in (a) of Figure 4. In a close-packed structure such as the face-centered-cubic (fcc), the energy required is

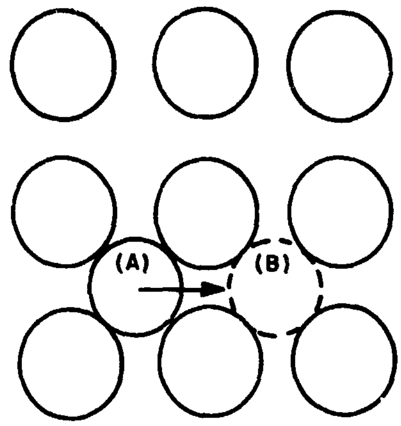


Figure 3. Face-Centered-Cubic Lattice (100) Plane Showing Interstitial Diffusion (From (a) to (b))

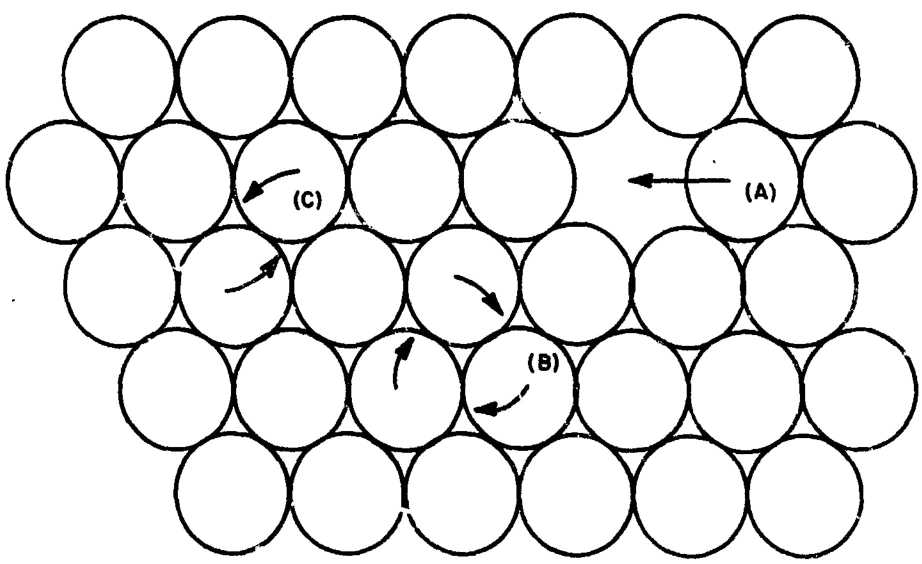


Figure 4. Schematic Representation, in a Close-packed Plane, of (a) Movement of an Atom into a Vacancy
(b) Three-atom-ring Interchange
(c) Two-atom-ring Interchange

nearly the same as that required to move interstitial atoms to neighboring interstitial positions. The rate of interstitial diffusion is greater than vacancy diffusion, however, because many more interstitial sites are available than vacancies. Of course, this requires the presence of interstitial atoms. The vacancy mechanism is the major means of diffusion in face-centered-cubic structures and is known to play a role in diffusion in other structures also. A vacancy mechanism should be dominant in pure metals and substitutional alloys.

(c) Interchange Mechanism - On an energetic basis, it is possible for diffusion to occur by the interchange of atoms. One form of this is the direct interchange of two atoms that simultaneously switch positions shown in (c) of Figure 4. When three or four atoms are involved, this process is described by a ring mechanism as shown in (b) of Figure 4. Although this mechanism is not known to be operative in metals, it has been suggested on theoretical grounds.

(3) Volume Diffusion. Volume diffusion is usually confined to interstitial, vacancy, and interchange mechanisms.[45] In interstitial alloys the interstitial mechanism is considered dominant. Diffusion in substitutional alloys is less readily described. On an energetic basis, however, a vacancy mechanism is probable. Diffusion of solute atom vacancy pairs may also occur. The energetics of diffusion point out the possibility of direct interchange of atoms by a ring mechanism as discussed previously. In close-packed structures, such as the face-centered-cubic, this mechanism is less favored than in the more open structure of body-centered-cubic crystals.

In a discussion of volume diffusion, Birchenall[34] concluded that interstitial or substitutional diffusion coefficients are much higher in body-centered-cubic phases than in face-centered-cubic structures when experiments have been performed with both phases. This conclusion requires that the data be extrapolated to the same temperature and that concentration differences not be too large. Second, it has been observed that where a solute decreases the liquidus temperature of an alloy, the diffusivity increases and vice versa. The activation energies of self-diffusion correlate roughly with the absolute melting points of many metals.

Experimentally, the diffusion coefficient (diffusivity) can be expressed as:

$$D = D_o \exp(-Q/RT).$$

D_o and Q vary with composition but are independent of temperature.

D_o is called the frequency factor, Q is called the activation energy for diffusion, R is the gas constant, and T is the absolute temperature.

(4) Other Types of Diffusion. Up to this point, only volume diffusion has been considered. Regions of high diffusivity, such as grain boundaries, surfaces, and dislocations, also contribute to the total diffusion in crystalline solids.

(a) Grain Boundary Diffusion - The quantitative influence of grain boundary diffusion is not known. Some studies of the effects of temperature and grain boundary structure have been made, however. For the metals investigated, grain boundary effects become more appreciable as the temperature is lowered. In silver, these influences become measurable at 1380°F and below. The explanation presented is that, within a boundary, the existence and formation of vacancies and the movement of atoms into these vacancies contribute to diffusion by being energetically favored. High angle boundaries display greater diffusivities than low angle boundaries because they are more disorganized; that is, in high angle boundaries, more vacancies exist and the interatomic bonding forces are lower than in low angle boundaries.[33]

(b) Dislocation diffusion - It has been shown that for silver, the total diffusivity is about 30 times the volume diffusivity in a perfect lattice at about one-half the absolute melting point. The remainder of the diffusivity is attributable to diffusion along dislocations. Thus, for temperatures below one-half the absolute melting point, the total diffusion coefficient is primarily determined by the dislocation density. The extrapolation of high temperature diffusivity data to low temperatures may not be of value for accurately determining the rate of atomic transport. If a solute is attracted to a dislocation, the apparent solute diffusivity can be significantly increased over solute diffusivity by volume diffusion.[31]

(c) Surface Diffusion - Surface diffusion by surface atoms of crystals, which show even greater diffusivities than atoms in grain boundaries or dislocations, may also occur. The weakly bound surface atoms require a lower activation energy for motion.

In the following few paragraphs, several articles that deal with diffusion phenomena are summarized and discussed. These articles present pertinent information regarding diffusion and its application to the solid state joining of metals.

During the initial stages of weld formation in diffusion welding, when the surfaces are being brought into contact, surface diffusion probably contributes to welding by assisting the filling of gaps in the interfacial region.[14, 46, 47, 48] Once in contact, volume diffusion by a vacancy mechanism overshadows other types of diffusion.[3, 47, 49, 50] Although the specific rates of grain boundary and surface diffusion are greater, the number of available atoms for these processes is smaller than for volume diffusion. Furthermore, although the energy required to move atoms by vacancy and interstitial mechanisms is nearly equal, the rate of vacancy diffusion is slower because there are fewer vacancy sites than interstitial sites. A restriction to interstitial diffusion is that it requires atoms having small radii. Vacancy diffusion can be strongly influenced by the method used for surface preparation. Cold working of the surface layers by a mechanical procedure raises the dislocation density near the surface. Consequently, the vacancy concentration near the surface will be increased by the attractive forces between vacancies and dislocations[32] and the formation of vacancies by the nonconservative motion of dislocations.[51] These processes will enhance volume diffusion by a vacancy mechanism. Pressure applied during welding promotes intimate contact between the pieces being joined and assists the formation of vacancies. The vacancies that are created may then participate in the diffusion process. Promotion of diffusion by the strain energy of cold work gradually decreases, due to annealing of the material at the welding temperature.

Studies of single-asperity welding of a gold needle to a gold plate[1, 52, 54] suggested that the area of contact and the breaking strength arose from three sources: (1) instantaneous adhesion, (2) weld area growth due to diffusion, and (3) weld area growth due to creep. The experimental results supported the theory that contact area growth at single asperities is primarily volume diffusion controlled.

In concluding this discussion on diffusion phenomena, it should be mentioned that, when alloy couples rather than pure metal couples are employed in diffusing systems, the situation becomes much more complex. The presence, initially, of intermediate phases introduces discontinuous concentration gradients. Growth of these and formation of new phases influences diffusion. Examples of this will be given later.

4. Grain Growth

When two clean surfaces are placed in intimate contact, a grain boundary is formed. The driving force for any boundary movement is the tendency to approach a minimum surface energy configuration. Surface tension will tend to reduce the boundary free energy by shortening the interface. The surface tension in crystalline solids is strongly dependent on orientation; close-packed planes have the lowest surface

tensions and are most likely to develop as interfaces.[54] The most stable grain configuration consists of hexagonally shaped grains meeting at 120-degree included angles.[29, 35] Forces between boundaries and impurities or voids will impede boundary motion and decrease the rate of grain growth regardless of grain size. It has been observed, however, that temperature alone may not provide sufficient energy to permit boundary migration and grain growth.[29] Processes not requiring a decrease in surface area were found to be more effective. Recrystallization, allotropic transformations, or a chemical potential (or activity) gradient across the interface are more influential. Parks[55] contends that recrystallization provides the mechanism of solid state welding; the welding temperature required is the lowest recrystallization temperature for that metal, approximately 0.4 of the absolute melting point. This mechanism also provides for intimate contact of the materials being joined because, as the recrystallization temperature is approached, the mean yield strength approaches zero.

Provided that a sufficient driving force is available, the presence of voids or impurities at the boundary will not prevent elimination of the interface and grain growth. A layer of voids or impurities, however, may remain at the original interface. Increased welding pressure promotes interfacial migration by suppressing void growth[56, 57, 58] and also increases the contact area available for diffusion. If impurities restrain movement of the boundary, pressure will have no effect on its migration.

Obviously, pressure promotes diffusion by increasing the surface area in contact. The influence of pressure on diffusion through defect formation is a more complex consideration. Plastic deformation near the interface and the associated movement of dislocations will create vacancies through nonconservative motion. Dislocations may also act as vacancy sinks, however, and can therfore contribute to the removal of voids.

Cunningham and Spretnak[58] state that boundary pinning by voids and impurities can be avoided by heating the specimens to the welding temperature prior to applying the welding pressure. In this way, boundary migration can proceed immediately after the interface is formed, before it can effectively be restrained by voids. Prevention of void formation can be facilitated by using smooth, clean surfaces and applying a reasonably low pressure to prevent void nucleation on impurities. Smooth surfaces also reduce void creation by permitting more intimate contact; the smaller open areas between asperities can thereby be more readily filled by surface diffusion. Occluded or adsorbed gases on the faying surfaces will also produce voids. Pore formation by the Kirkendall

mechanism, as described later, may occur during dissimilar metal welding. Machinability of the metal may also effect pore formation by supplying or preventing surface cracks, tears, and other defects during preparation. Vacancy sinks (moving grain boundaries, dislocations, and subgrain boundaries) may effectively reduce the void volume. The efficiency of vacancy absorption by these defect structures is a function of the metal under consideration.

In addition to recrystallization and grain growth, polygonization may occur if a sufficient number of dislocations are present. These dislocations may be introduced either prior to or during bonding. It was shown that a greatly deformed region having a high dislocation density existed near the bond interface and resulted from the welding process.[29,58,59]

Williams and Bever[60] suggest that regions of oxide at the interface between aluminum and copper tend to spheroidize and be absorbed by the base metals during elevated temperature diffusional treatments. Precipitation of dissolved oxide may occur at a later stage.

It is doubtful that recrystallization is required for diffusion welding. Refractory metals have been joined without evidence of recrystallization.[2,13,47,61] Recrystallization is a "driving force" that may hasten the formation of diffusion welds, that is, influence the kinetics of metallic bond formation. This may be particularly effective when other forces are not available to promote welding. Chemical concentration gradients (actually chemical activity gradients) and phase transformations are also capable of promoting welding. It has been observed that moving boundaries assist the removal of lattice defects, particularly vacancies and voids. In a corrosive environment, the presence of an interface (a high energy region) at the joint may accelerate the corrosion and drastically shorten the life of the joint. For such a situation, removal of the interface by recrystallization and grain growth across the interface would probably be desirable.

One of the advantages of high temperature welding is that, for those metals in which the oxide is soluble (e.g., Fe and Cu), dissolution of the oxide layer may then assist in weld formation.[48] Reviewing previous work, the authors suggest, as others have, that in low deformation welds, volume diffusion is the rate determining process for weld formation. This view is supported by the numerous reports of joint strength increases in room temperature deformation welds, which were subsequently heat treated. Diffusion, recovery, recrystallization, grain coalescence and growth, spheroidizing of lenticular defects, and reduction of elastic recovery effects all probably play a role; however,

void formation by the Kirkendall mechanism and formation of brittle intermetallic compounds may produce deleterious effects. Heat treatment of deformation welds in copper gave results similar to those of Cunningham, et al., described earlier.[58] Discontinuities initially present after welding spheroidized, coalesced and finally disappeared after recrystallization and grain growth had occurred. Heat treatments using copper specimens were conducted from 750° to 1650°F for up to 167 hours.

Similar results were observed for aluminum deformation welds; however, the insoluble alumina, present at the interface, prevented void formation as well as grain growth across the interface. The increase in weld strengths occurred with no observable changes in microstructure or material hardness and was attributed to atomic rearrangement at the interface.

At low deformations, the extent of metallic contact is small. Thermally activated surface diffusion along gaps or through vacant lattice sites increases the welded area.

Heat treatments applied to mild (0.08 percent C) and decarburized steel suggested that elastic recovery effects have, in the past, been overemphasized. The weld strengths of specimens heated to above 750°F while under some fraction of the initial welding load exceeded those of specimens heat treated under no load. With no applied load, heat treatment would only provide stress relief on a microscale. Under load, interfacial deformation and subsequent stress relief could occur with no overall reduction in thickness at the weld, and thereby improve the weld strength.

It can be seen, therefore, that grain boundary movement and grain growth are influenced by a number of factors. Those factors that tend to promote grain growth include:

1) Surface tension.
2) Increased temperature.
3) Increased pressure.
4) Recrystallization.
5) Phase transformations.
6) Chemical potential gradients.

Voids, impurities, and compounds formed during diffusion have the opposite effect.

5. Dissimilar Metal Welding

The following will be devoted to a discussion of some general factors to be considered when diffusion welding dissimilar metals. Following this, several references are summarized which provide pertinent information and reveal aspects of the problem which may be encountered in practice.

Diffusion across a dissimilar metal interface can result in the formation of voids by the Kirkendall mechanism. This effect is explained by vacancy diffusion of species having unequal diffusion coefficients. The greater mass flow of atoms in one direction is balanced by a flow of vacancies in the opposite direction. Although most of the vacancies will be absorbed by dislocations, pores can form from excess vacancies. Coalescence of vacancies to form voids and growth of these voids by continued vacancy absorption is likely. A driving force for this is reduction of surface energy by formation of spheres having a low surface-area-to-volume ratio. This phenomenon has been examined in some detail.[29, 30, 58, 62]

In the joining of dissimilar metals by diffusion welding, there are two principal factors which must be considered: (1) diffusion reactions that may occur at the interface and cause brittle compound formation or Kirkendall porosity and (2) differences in thermal expansion coefficients that may fracture previously formed welds upon cooling. Depending on the system being joined, it may be desirable to employ one or more intermediate materials to prevent undesirable reactions, to provide a gradation in expansion coefficients, or both. The use of intermediate materials is discussed in the next section of this report.

In any dissimilar metal joint, chemical activity gradients exist. Arising from concentration gradients or differences in solid solubility, diffusion generally occurs in the direction of lower concentration. An activity gradient in the opposite direction, however, may cause diffusion to occur against the concentration gradient. A certain amount of energy is required to activate the diffusion process. In a study of the diffusion welding of dissimilar ferrous alloys,[63] the interstitial diffusion of C and substitutional diffusion of Fe, Cr, and Ni were shown to be strongly interdependent. For example, diffusion of Ni and Cr from austenite into ferrite and precipitation of carbides in the austenite will alter the solubility of C in the matrix and will change the partition of C between the matrix and precipitated phases. This, and the varying solubility of C in each phase, will alter the diffusion rates. Precipitation of Cr carbides will reduce the removal of Cr from the austenite by diffusion. Thus, complex reactions can occur depending on the system in question. A simple analysis of these occurrences cannot always be formulated.

The behavior of dissimilar metal couples depends largely on the alloying interactions that can occur among the components present. Miscible systems will usually weld readily but may develop porosity at elevated temperatures (Cu-Ni and Ni-Fe). Couples forming intermetallic compounds can produce divergent behavior, depending on the nature of the compounds created. Brittle intermetallic layers have been found to be detrimental to a weld when thick but had little influence when thin. If the compound was ductile, the weld was strong, independent of thickness, and failure occurred in the weaker metal. These data were obtained during roll welding studies; however, the influence of diffusional processes should pertain to other solid state joining techniques where gross deformation is absent.[64]

The presence of several intermetallic phases in a binary system does not insure that zones of each will be formed during diffusion welding. In a study of the Al-Zr system,[65] it was observed that only one, $ZrAl_3$, out of a possible nine intermediate phases, formed at elevated temperatures (1020° to 1200°F) and extended times (1 to 150 hours). The rapid growth of the $ZrAl_3$ phase was related to the extremely rapid diffusion of Zr and Al in that phase. This diffusional flux was, in turn, related to the atomic structure of the phase, which also accounted for the presence of lenticular voids at the $ZrAl_3$-Al interface as well as spherical voids within the $ZrAl_3$. Even if a number of intermediate phases are stable at the temperature in question, as indicated by the pertinent equilibrium phase diagram, the temperature must be high enough and the time long enough for nucleation and growth of these phases to occur.

In the diffusion welding of butted cylinders of Al and Ni in a die so that hydrostatic pressures were generated,[16] the decrease from the maximum in the curve of weld strength versus temperature, shown in Figure 5, signifies the initiation of the formation of brittle alloy layers. If the strength value rises again at a higher temperature, a new or modified intermetallic may have formed at the interface. Similar effects are observed for weld strength versus time as shown in Figure 6. Couples of aluminum with copper, iron, or zirconium showed, in general, the same phenomena as aluminum and nickel. For all four dissimilar metal couples, pressure tended to raise weld strength as shown in Figure 7. Strength decreases were attributed to compressive fracture of a brittle zone that had formed, or time and temperature effects.

Intermetallic zone thickness was found to be controllable by pressure. Increased pressure decreased penetration for the Al-Cu and Al-Ni systems. The reverse was observed in the Al-Fe system. Intermetallics that decrease in volume during formation were seen to be

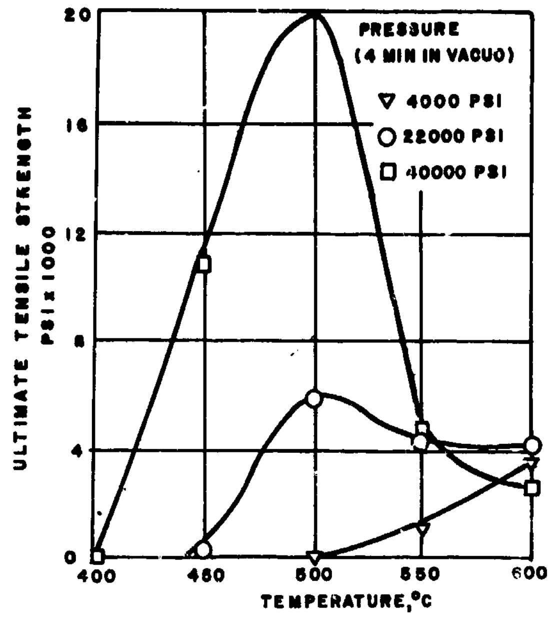


Figure 5. Effect of Temperature on Ultimate Tensile Strength of Al-Ni Couples Welded at Various Pressures[16]

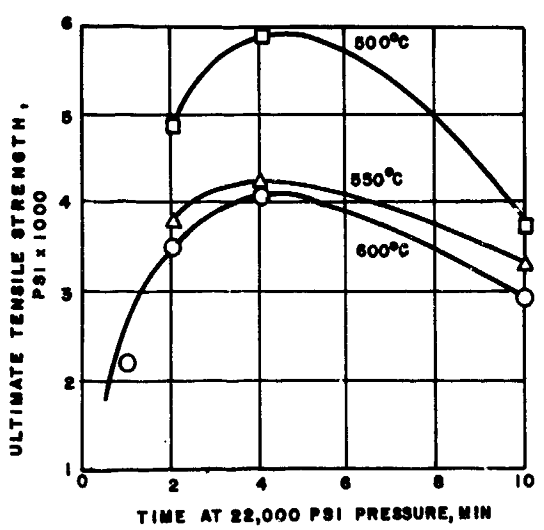


Figure 6. Effect of Time at Pressure of 22,000 psi on Ultimate Strength of Al-Ni Couples Welded at Various Temperatures[16]

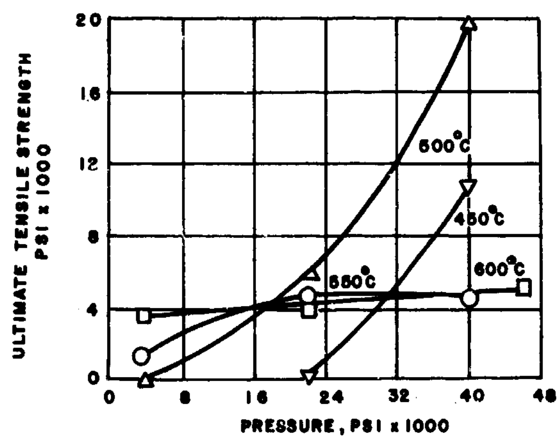


Figure 7. Effect of Welding Pressure on Ultimate Strength of Al-Ni Couples Held 4 Minutes in Vacuo at Various Temperatures[16]

reduced by increasing pressure while those which expanded during formation increased in thickness with the application of additional pressure. The authors also investigated the welding of aluminum to copper and to zirconium.[66]

In another study of diffusion welding Al and Ni, Castleman and Seigle identified the two diffusion layers that were observed as Ni_2Al_3 and $NiAl_3$ phases.[67, 68] Disks of the high purity metals had been pressed together in vacuum for various times and temperatures. Temperatures of 752° to 1157°F permitted the visable formation of the phases given above but not other intermetallic phases presented in the Al-Ni equilibrium diagram. Above a certain thickness, the Ni_2Al_3 phase grows by volume diffusion. Increased pressure retards its growth by reducing the chemical diffusion coefficient. Similar conclusions were reached with regard to the $NiAl_3$ phase although increased pressure exerted a smaller influence on its growth. Pressure induced layer growth of UAl_3 in the U-Al system, on the other hand, was explained by a purely mechanical effect. Pressure decreased the tendency for macroscopic defect formation and increased the cross-sectional area available for interdiffusion.[67, 68]

6. Intermediate Materials

Although not a part of the mechanism of diffusion welding, the use of intermediate materials in diffusion welding is of considerable importance. Intermediate materials can be used to:

1) Promote diffusion at much lower temperatures than for self-welding.

2) Promote plastic flow and surface conformance at lower pressures.

3) Prevent the formation of intermetallic compounds.

4) Obtain clean surfaces.

5) Reduce effects due to differences in thermal expansion coefficients.

Intermediate materials are commonly used in either the form of foil or as electroplates. Foreign atom diffusion rates are generally much greater than self diffusion rates. Thus, the intermediate layer can accelerate the diffusion stage in the welding process. Intermediate materials with low yield strengths compared with the base metal permit larger contact areas for a given applied pressure. In systems which form intermetallic compounds, an intermediate layer can be used to

restrict the interdiffusion of the components to be joined and thus prevent the creation of brittle compounds. For metals such as aluminum, which are difficult to clean for welding, an electroplate of a more readily cleaned metal can be used to facilitate welding.

Excessive diffusion may produce voids by coalescence of microvoids not detectable in the as-welded structure, or by expansion of trapped gas, or as a result of the Kirkendall effect. In addition to changes in the weld structure, extensive diffusion into the base metal may occur, promoting recrystallization at a lower than normal temperature, extensive grain growth, formation of intermetallic phases, segregation of impurities at grain boundaries, or changes in any number of physical or mechanical properties which are related to composition and structure.

In some cases, therefore, a diffusion aid should not be used. The distinct possibility exists, however, that such an aid will be required to achieve satisfactory welding. To accomplish this, the intermediate material should preferably form and remain in solid solution with the base metal, should diffuse rapidly, and should be applied in thin layers to the faying surfaces.[14,15,69,70] The formation of a dilute solid solution of the intermediate metal atoms in the base metal, near the interfacial region, is probably desirable. If the solubility of the diffusing element decreases with decreasing temperature and its concentration in the base metal is near the solubility limit, intermetallic compounds can be precipitated upon cooling.[70]

The importance of the factors that can influence diffusion are probably dependent on the particular system being considered. Atomic sizes, valences, melting points, and solubility limits are all involved[14] although they are not independent. Solubility has been related to the other factors.[70]

In a study of diffusion welding of iron, cobalt, and nickel base, high temperature alloys using an intermediate having a small atomic diameter (beryllium), it was suggested that vacancy formation could aid weld formation.[15] Because the beryllium atoms in the intermediate move (by a vacancy mechanism) more rapidly than the base metal atoms of Fe, Co, Ni, Cr, W, or Mo, vacancies will be created at the interface. These vacancies permit the base metal atoms to move readily (also by a vacancy mechanism) through the interfacial region. As the beryllium diffuses away from the interface, its diffusivity is expected to increase because it causes melting point depression when added to Co, Cr, Fe, and Ni. This occurrence was discussed earlier in the area of this report dealing with diffusion.

A disadvantage of using thick, soft intermediates is that, at elevated service temperatures, the joint will weaken considerably. This effect can be minimized by reducing the thickness of the intermediate layer. A ductile intermediate promotes intimate surface contact, however, and this may more than compensate for its low strength.[72] If a soft intermediate is relatively thick, the situation is similar to brazed joints having thick but weak filler alloy between the parts being joined. In brazed butt joints it has been shown that the tensile strength of the joint decreases with increasing thickness of the filler alloy, as shown in Figure 8. The maximum joint strength, however, may be considerably greater than the strength of the filler alone because of the plastic restraint exerted by the base metals being joined.[73] Alloying of the filler metal and base metal is not always required for sound, brazed joint formation. In the silver brazing of iron base alloys, silver and iron are nearly insoluble in each other.

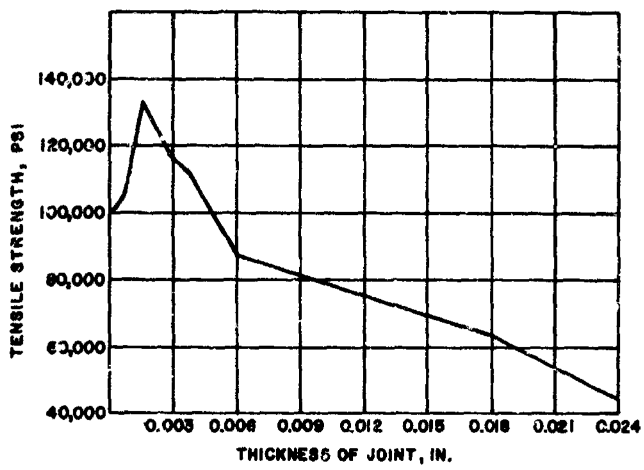


Figure 8. Relationship of Tensile Strength to Joint Clearance of Ag-Cu-Cd-Zn Brazed Joints in Stainless Steel[74]

Because clean faying surfaces facilitate diffusion welding, an intermediate can be used as a plated layer, for example, to protect the underlying base metal from contamination. The intermediate can provide a clean surface for welding. A relatively noble or oxidation resistant metal (e. g., Cu, Ag, or Au) would be desirable. Metals that dissolve their oxides at the welding temperature or form oxides that are unstable at the welding temperature will promote diffusion welding by aiding the dispersal of these contaminants.

In view of the phenomena involved in the use of intermediates, a wide choice of the method for their use is available. To obtain the desired joint properties between either similar or dissimilar metal combinations, single or multiple layered intermediate forms may be used.

A consideration that must not be overlooked in developing systems using intermediate metals to promote diffusion welding is that of corrosion. The service environment and the chemical characteristics of the metals themselves (e.g., position in the galvanic series) are primary aspects to be considered.

Section III. PROCESSING

The major diffusion welding techniques are discussed in this section. Methods of pressure application, heating, atmosphere selection, and cleaning are presented. Roll welding is also described, including some background information.

Structural joints that may be fabricated by diffusion welding and roll welding are presented.

As shown in the discussion, solid state diffusion welding possesses several important advantages over other joining methods. However, several disadvantages are inherent in all the diffusion welding techniques. These include the following:

1) Long welding times are generally required in addition to the time necessary for heating and cooling.

2) Long welding times at elevated temperatures can cause excessive creep or the materials being joined.

3) The total time, including heating, welding, and cooling, may induce metallurgical changes in the materials being joined.

4) Processing costs increase rapidly with increasing requirements for welding tempetature, welding pressure, and part size.

5) Open structures, such as honeycomb or truss core, cannot be fabricated without supporting material.

For some applications it may be advantageous to initially weld simply shaped specimens followed by a final forming or machining operation. A requirement for a curved or specially shaped joint having the same properties as the base materials would suggest the use of this procedure.

1. Processing Methods for Diffusion Welding

There are three major processing methods used for the production of diffusion welded joints. These are press welding, differential-thermal expansion welding, and gas pressure bonding. The primary differences between these methods are the means by which pressure is applied to the pieces to be joined. In the discussion that follows, each technique will be briefly described and its advantages and disadvantages mentioned. The election of a particular welding method depends to a large extent on the materials being joined, the size and shape of the part, and the configuration of the joint.

a. Welding Pressure

The three major diffusion welding methods apply the welding pressure by different principles. As discussed earlier in the report, the purpose of the welding pressure is to bring the surfaces into intimate contact such that diffusion can provide atomic transport across the interface. The pressure required for welding is primarily dependent on the materials involved, their condition (annealed, cold worked, surface roughness, etc.), the welding temperature, and the welding time (creep effects can become important at long welding times and high temperatures).

(1) Press Welding. This pressure application method consists of placing the parts to be joined between the platens of a press (hydraulic or pneumatic). The platens may support appropriately shaped dies that will apply the welding pressure to the faying surfaces. This method is well suited for joining flat pieces or parts having parallel, opposite faces such as lap joints, sandwich composites, and butted rods or tubes. Butt joining of bars or tubes is frequently performed within a sleeve in order to prevent lateral deformation. An internal mandrel may also be employed when joining tubes as shown in Figure 9. This arrangement permits the approach to isostatic conditions. The advantages of press welding include:

1) The equipment and welding procedure may, depending upon the pieces to be joined, be relatively simple.

2) The largest part of the research on diffusion and deformation welding has been conducted using press welding methods.

3) The load applied to the joint can be precisely measured using a load cell installed in the press.

4) Pressure control is precise, provided that the joint area is not too large (possibly up to 6 feet by 6 feet with commercially available units).

5) Powder compaction and simultaneous joining to solid bodies is feasible.

6) A wide range of welding temperatures may be used.

Disadvantages of press welding are:

1) Large joint areas introduce problems of precise pressure and temperature control.

2) Brittle materials are difficult to join without proper support because isostatic pressures are not usually achieved.

3) Welding across complex shaped or angular surfaces is not readily accomplished.

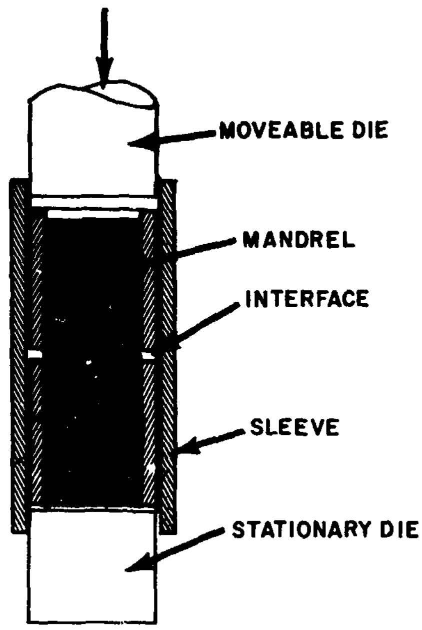


Figure 9. Press Welding a Tube Using a Sleeve and Mandrel

(2) Differential-Thermal Expansion Welding. In this method the welding pressure results from the difference of thermal-expansion coefficients of the pieces to be joined and a supporting fixture or just the pieces to be joined themselves. A relatively small number of the reports surveyed gave information on this technique although it has been used.[3, 46, 44, 50, 75, 76, 77] For butting rods, tubes, or flat plates, an arrangement such as that shown in Figure 10 may be used. The fixture must have a lower expansion coefficient than the pieces to be joined.

Joining overlapped tubes by this method can be performed as shown in Figure 11. A mandrel with a higher expansion coefficient and a retaining ring with a lower coefficient than the parts to be joined can provide sufficient welding pressure. The specimens can be assembled at room temperature using interference fits in order to raise the maximum pressure obtainable. This technique is currently being employed to join 8-inch diameter rings of 2219 aluminum to Type 321 stainless steel.[76]

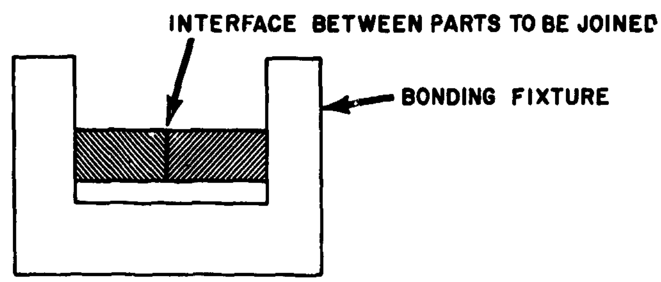


Figure 10. Welding by Differential-Thermal Expansion

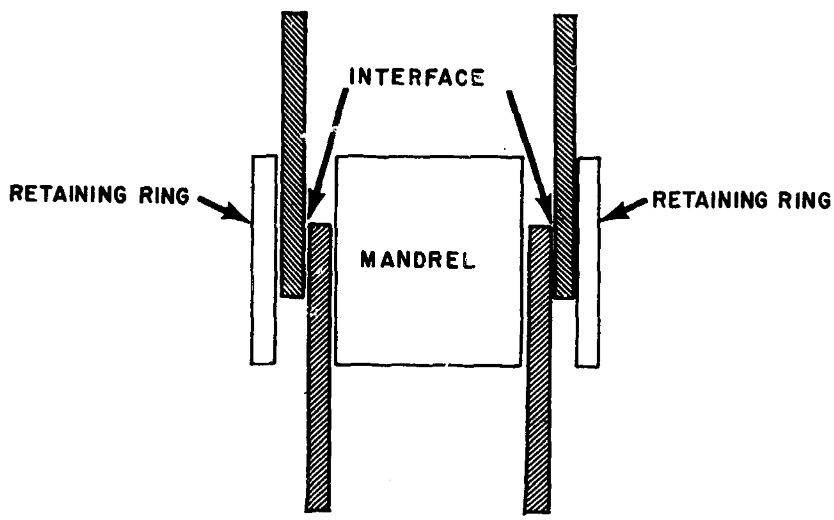


Figure 11. Joining Overlapped Tubes by Differential-Thermal-Expansion Welding

The advantages of Differential-Thermal Expansion Welding are:

1) The ability to join pieces is less dependent on part size than with other methods.

2) Compared with the other joining methods, the equipment requirements can be very inexpensive, depending on the size and complexity of the joint.

Disadvantages of differential-thermal expansion welding include:

1) Precise measurement and control of welding pressure is difficult.

2) A fixture material having the proper expansion coefficient and adequate strength at the welding temperature may not always be available for the joint configuration and materials being considered.

(3) Gas-Pressure Bonding. In the gas-pressure bonding process, the components to be joined are fabricated or machined to final size, cleaned, and assembled into an expendable container or are edge-welded to produce a pressure-tight evacuated envelope. The assembled components are heated to an elevated temperature in an autoclave containing an inert gas at high pressure as shown in Figure 12. The isostatic pressure is uniformly transmitted (through the container, if one is used) and forces all of the mating surfaces into intimate contact along any desired surface contour. The mating surfaces are held under pressure at temperature for a sufficient time to permit solid state welding between the components.

Cold wall autoclaves containing a resistance heater are used to attain the high gas pressures and temperatures needed for gas pressure bonding. Specimens to be welded are inserted in the heater, which is insulated from the autoclave heads and main body to prevent excessive heating of the vessel. In autoclaves currently in use, it is possible to exert a hydrostatic gas pressure of 10,000 psi at temperatures up to 3000°F. Times of about 1 to 5 hours are normally used. Helium or argon is usually used as the pressurizing gas. The maximum operating pressure of the equipment is generally used in order that a lower welding temperature or a minimum of time can be used, which may be advantageous in controlling grain growth or diffusion.

In welding studies accomplished to date, it has been found necessary to evacuate the welding can prior to gas pressure bonding. The residual gas, otherwise, is compressed into the weld interfaces and interferes with the welding operation. For nonreactive materials, such as stainless steel, a pressure of 25 to 50 microns is adequate. More reactive materials, such as beryllium, may require pressures substantially below 1 micron to insure clean welds.

The cycle time usually required, that is loading, heating, cooling, and unloading, is approximately 24 hours. The life of most of the components is indefinite with only nominal maintenance costs (vessel,

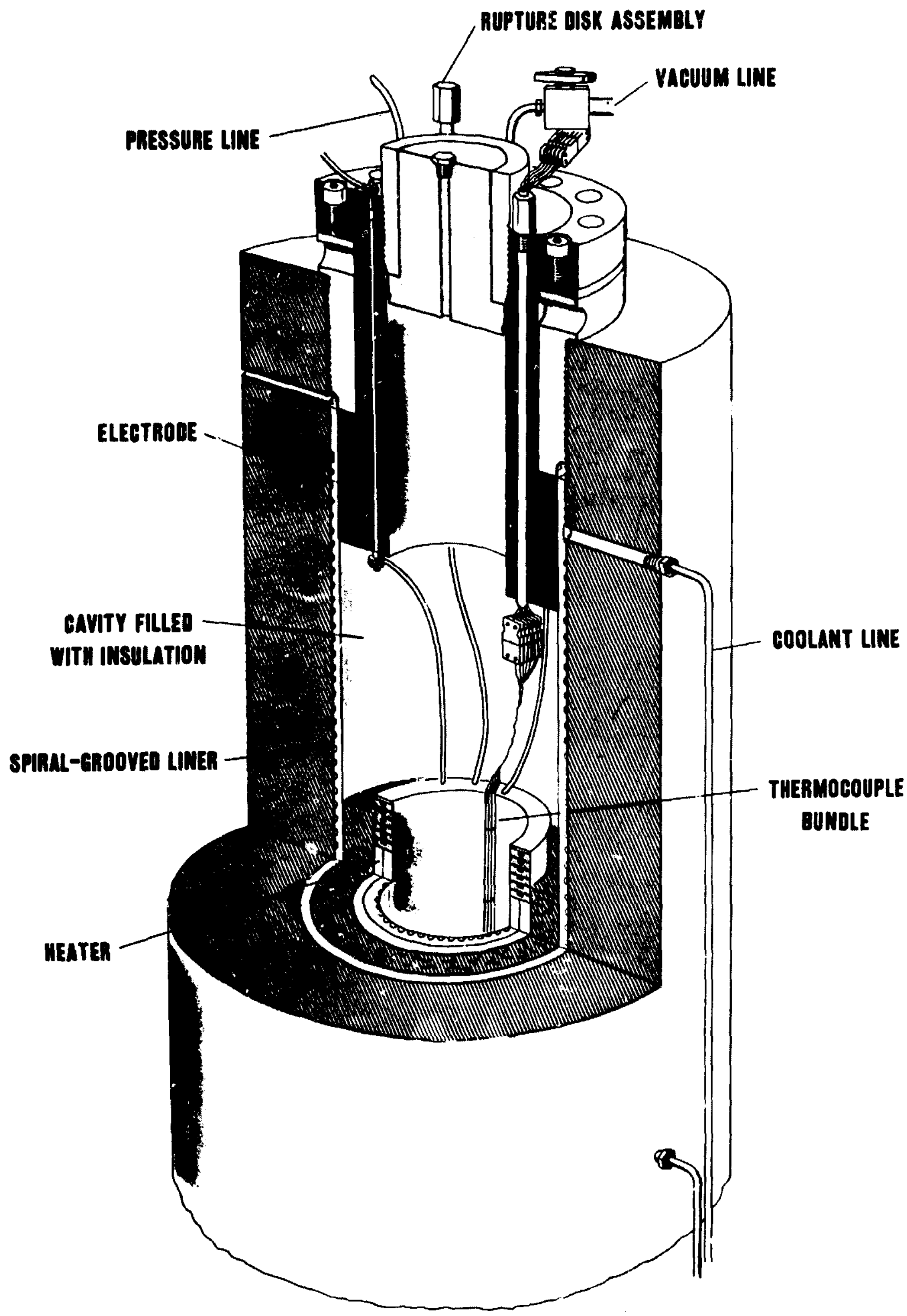


Figure 12. Sectional View of Cold-Wall High-Pressure Autoclave[2]

compressor, and control equipment). The life of the furnace depends to a large extent on the temperatures generated - at least 75 cycles up to 1500°F and 15 cycles to 2100°F.

Numerous descriptions of this type of equipment have appeared in the referenced literature.[2, 11, 13, 72, 78, 79, 80, 81] In all cases, the equipment is similar in design, differing only in size and maximum capabilities.

This joining method possesses the following advantages:

1) Brittle materials, such as oxides and hydrides, can be clad with ductile or brittle materials without cracking of the materials, because isostatic pressures are used.

2) Ceramic, metallic, cermet, and dispersion powders can be readily compacted to very high densities without stringering or cracking and can be simultaneously welded to other solid pieces.

3) Complex shapes can be produced from many similar or dissimilar materials, usually in a one step operation.

The gas-pressure bonding method also possesses several disadvantages:

1) The envelopes used to contain the parts to be joined must be carefully welded so that they are leak free. Any leaks that are present during welding will, cause the welding pressure to approach zero, and contaminate the parts being welded.

2) The part size is limited by the furnace and autoclave sizes. Increasing the sizes of the furnaces and autoclaves can be accomplished, but the equipment costs rise rapidly with capacity.

b. Welding Temperature

Heating methods seldom provide significant problems in diffusion welding. The most common methods include radiant, resistance, and induction techniques. The method selected largely depends on the welding technique, the temperature and time of welding required, and the size and shape of the parts to be joined. With large pieces, uniformity of temperature may be difficult to achieve. Induction heating frequently permits more rapid attainment of the welding temperature than the others.

c. Welding Atmosphere

Air, inert gas, and vacuum are commonly used atmospheres. Occasionally active atmospheres such as hydrogen are used. Gas-pressure bonding requires an evacuated and sealed container. Whenever possible, vacuum is desirable because it aids in the prevention of undue surface contamination and promotes the removal of sorbed surface impurities.[7] This is particularly important when joining reactive metals (Be, Ti, Zr) and the refractory metals (Cb, Mo, Ta, W), which are readily contaminated and their properties impaired by reaction with active gases.

Welding in an inert gas environment requires either vacuum or displacement purging apparatus as well as equipment to dry the gas. An inert gas is suitable, provided that it does not permit recontamination of the faying surfaces. When some materials, such as plain carbon steels, are welded, active atmospheres promote surface cleanliness by the reduction of oxides.

By canning the parts to be joined or performing the welding operation in a suitable chamber, inert atmosphere or vacuum can be utilized by all three of the methods used for pressure application, which were discussed earlier.

2. Roll Welding

Although it is not a diffusion welding technique, roll welding is included in this report because of its potential usefulness for aerospace manufacturing. In the discussion that follows, some of the important roll welding studies are described in order to provide some background for a better understanding of the process. The major factor that differentiates roll welding from diffusion welding is the use of rather large deformations (possibly as high as 95 percent) during the rolling operation. Much of the theory given earlier for diffusion welding is equally applicable to roll welding.

Although some specific information is given on roll welding parameters, particularly for Al and Ti, data concerning the roll welding of similar and dissimilar combinations of Be, Al, and Type 321 stainless steel have not been included. Although the roll welding literature was not completely surveyed, the information presented should be helpful in the development of roll welding techniques for specific applications.

In the roll welding process, the sh to be joined are given a suitable surface preparation and placed t... ether for rolling. The actual rolling process may be carried out hot or cold depending on the mater-rials to be joined. The reduction required during rolling depends on the materals, the rolling conditions, and the surf...ce preparation used.[10] It has been observed experimentally that a threshold deformation must be exceeded before any welding will occur (about 40 percent for commercial aluminum rolled in air at room temperature). The threshold deformation and the weld strength are the major parameters against which other factors in the roll welding process are evaluated. Factors that tend to reduce the threshold deformation include:

1) Lower melting point of the material.
2) Increasing rolling temperature.
3) Cleaner surfaces.
4) Rolling cubic - rather than hexagonal-structured metals.
5) Increased oxygen solubility of the base metals.
6) Decreased roll speed.
7) Relative movement of the pieces across the weld interface.

Effects due to metal purity, prior cold work, and post-heat treatment of the welds have been found to vary with the system in question. During rolling of dissimilar metals, differences in thermal expansion coefficients must be considered.

In the discussion of a mechanism of roll welding, several factors have been found to be important. The breakup of surface oxides and the creation of extended metallic contact, overcoming an energy barrier in order to reorient surface atoms or disperse oxides, and the deleterious effects of retained elastic stresses are believed to be the dominant factors. The nature of real surfaces and their progressive contact under pressure have been discussed previously and will not be repeated here.

Milner, et al.,[10, 43, 64, 82, 83, 84, 85] have investigated the roll welding technique in which two metals were bolted together and compressively rolled to some predetermined degree of deformation. In effect, the rolling process served to expand the interface in the absence of contaminant gases to a degree in which metal-metal atoms were brought into contact. In a discussion of the mechanism[84] of roll welding, the effect of the variables of surface preparation, surface contamination, roll pressure, time and temperature were considered.

Experimentally, 4 x 4-inch metal composites were passed through compressive rolls, and regions of the welded couple were tested in tensile shear after the amount of deformation had been calculated. The effect of adsorbed contaminants was evaluated by applying various surface preparations and measuring the shear strength of the composites as a function of percent deformation. The preparations included machining, scratch brushing followed by degreasing, degreasing followed by scratch brushing, and electropolishing the surfaces. In one case, aluminum specimens were preheated to 932°F and cooled in a desiccator to prevent readsorption of impurities, while in the other, this preliminary step was omitted. The results indicated that for unheated samples degreasing followed by scratch brushing was superior; the shear strength for a given deformation was greater than for the other preparations. The minimum deformation (the threshold deformation) required to produce a measurable weld strength (40 percent for aluminum) was lower, as shown in Figure 13. The heated specimens, electropolished or machined, displayed higher weld strengths than those not preheated but not as high as scratch brushed composites. The authors attribute the effect of heating and cooling in a desiccator to the removal of adsorbed contaminants. Baking out the specimens in air or vacuum (5 x 10^{-3} mm Hg) to as high as 1112°F indicated that the bake-out atmosphere (air or vacuum) had little influence on weld strength at a constant 60 percent deformation, although the higher temperatures produced greater weld strengths. Bake-out at 1112°F and 60 percent deformation produced a weld shear strength (4.5 tons per square inch) less than that of similarly prepared but scratch brushed aluminum specimens (6 tons per square inch), implying factors other than contamination play an important role.

Samples of anodized aluminum (thick oxide layer) would not weld even with 80 percent deformation. If, however, these were baked at 932°F and cooled in a desiccator, they displayed a good weld above a threshold deformation of about 50 percent, but not as good as that achieved after scratch brushing. Thick oxides do not completely deform as one, as illustrated by microscopic examination, while thin oxides do after scratch brushing. Thick oxides, therefore, result in a lower percentage area of metal-to-metal contact and a lower strength. Similarly, it was observed that the other sample preparations, without final scratch brushing, produced thin oxide layers that broke up independently and reduced the area of metallic aluminum contact. Parallel studies of surface preparation and oxide formation were made using copper to correlate with the results on aluminum. Generally, the same results were obtained. However, with thick copper oxide layers, the threshold deformation was about 60 percent compared to about 45 percent for a scratch brushed copper couple, a difference of 15 percent. This same difference for anodized versus scratch brushed aluminum was about 10

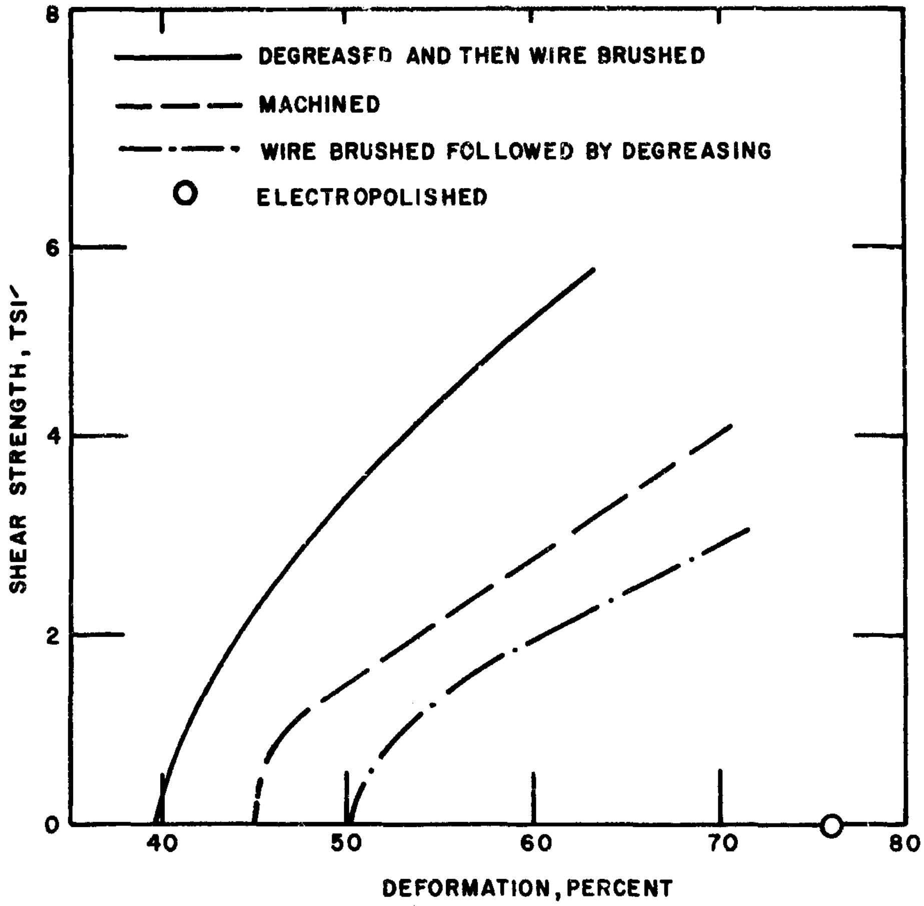


Figure 13. Effect of Surface Preparation on the Roll Welding of Aluminum[84]

percent. The greater change in the threshold deformations of copper couples was ascribed to more irregular fracture characteristics of massive copper oxide. This reduced the area of metallic copper contact to a greater extent than the metallic aluminum contact. On the anodized aluminum, the thick oxide fractured into separate, rectangular blocks. When the interfacial oxides broke up as a single layer, metallographic inspection showed that the total length of the fractured oxide particles along the interface equalled the original length of the specimens. Weld-strength data for aluminum are given in Figure 14.

In another study,[83] the authors examined the effect of temperature. The specimens were cleaned, scratch brushed, and seam welded along the outside edge of the composites to prevent high temperature oxidation. Weld strength, as a function of deformation, was determined for aluminum, Armco iron, magnesium, and zinc. For aluminum, the threshold

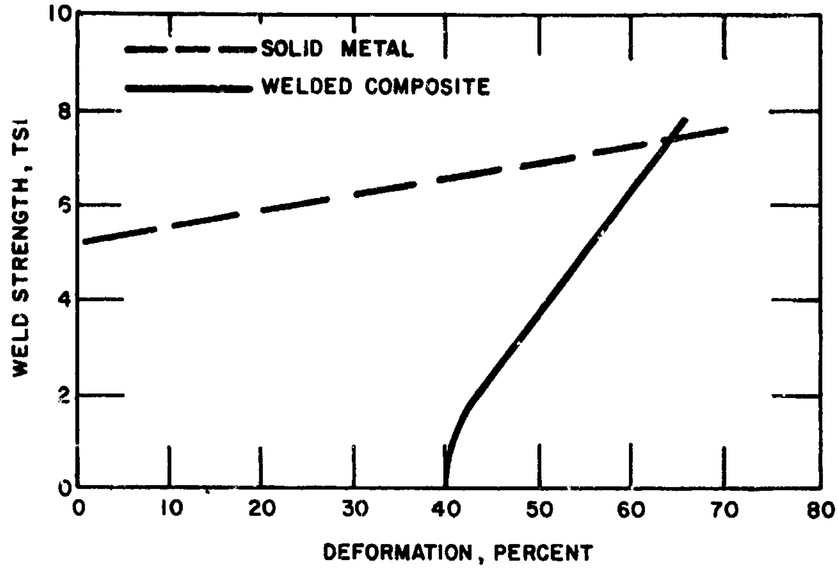


Figure 14. Weld Strength as a Function of Deformation for Roll Welded Aluminum[85]

deformation decreased from 40 percent at room temperature to 5 percent at 1112°F, as shown in Figure 15. Further, at room temperature, the oxide broke up with the work-hardened, scratch brushed layer, and welding of relatively soft bulk aluminum occurred; whereas, at elevated temperatures, welding occurred between the work hardened surfaces resulting in higher shear strengths. Armco iron achieved a weld strength equal to the strength of the solid metal at 1652°F and 14 percent deformation and metallographically displayed recrystallization and grain growth of the scratch brushed region during the heating period prior to rolling. Inexplicably low weld strengths were developed with magnesium between 752° and 1112°F. Similar results were obtained with zinc as with magnesium, and at room temperature the scratch brushed layers displayed brittleness in some regions and ductility in others. The authors suggest that the weaker welds of the hexagonal metals, magnesium and zinc, is due to independent breakup of the oxide layer. This independent fracture tendency is related, by the authors, to relative motion of the surfaces during rolling caused by the strongly orientation dependent deformation behavior of the hexagonal metal structures. The threshold deformations for several metals are given in Table II.

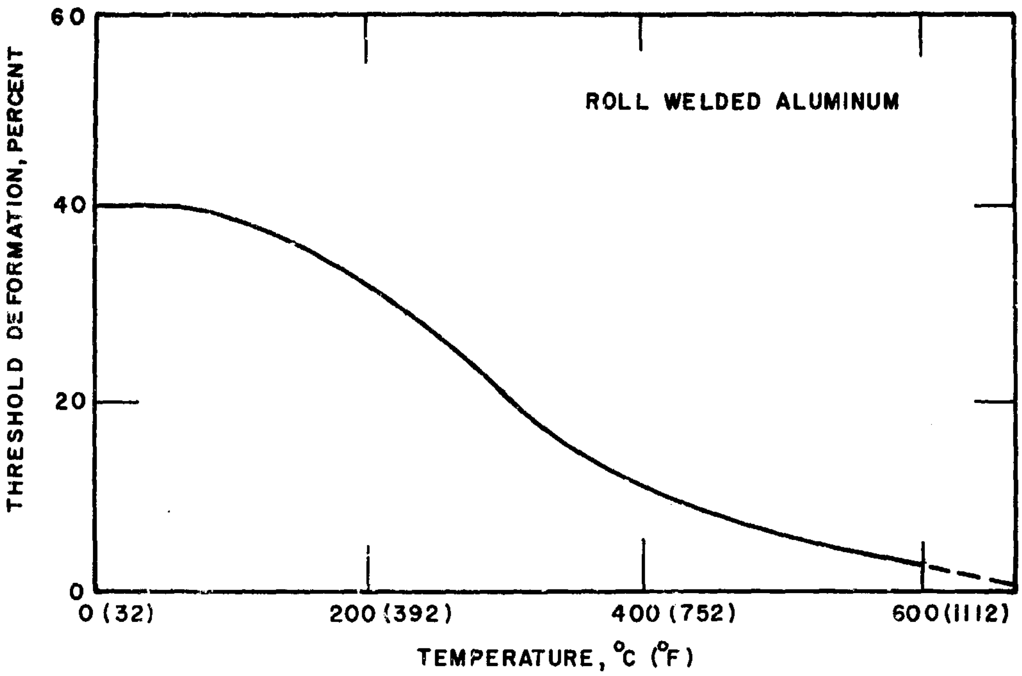


Figure 15. Variation of Threshold Deformation with Temperature for Roll Welded Aluminum

Table II. Threshold Deformations for Room Temperature Deformation Welding

Metal	Melting Point, °F	Hardness, BHN Annealed	Hardness, BHN Cold-Worked	Threshold Deformation, percent
Tin	450	5	6	15
Lead	621	4	4	10
Zinc	787	30	35	55
Aluminum (super purity)	1220	16	28	25
Aluminum (commercial purity)	-	20	40	40
Copper	1981	30	100	45

It is suggested that the ease of solid state welding is related to atomic mobility, and therefore to a reduced or homologous temperature [ratio of a welding temperature (°K) to the melting temperature, T_m (°K)]. For the metals studied, the threshold deformation decreased with increasing temperature until about one-half of the absolute melting point (0.5 T_m); whereas, for the scratch brushed cubic metals, the threshold deformation decreased rapidly with further increase in temperature. This follows from the fact that above 0.5 T_m, fine grained and ductile layers welded. This behavior was not observed with the hexagonal metals.

In a study of roll welding of dissimilar metals,[64] Milner et al, reviewed their earlier work and injected the metallurgical variable into their studies. It was found that, for single metal composites, longer welding times (lower roll speeds), greater oxygen solubilities of the metals at elevated temperatures (effectively increasing the area of metallic juncture), and increased metal purity all enhanced the weld strength. Heat treatment after rolling generally improved the weld, although, with magnesium and silver, the weld was inexplicably destroyed by this procedure. When dissimilar metals were roll welded, relative movement occurred at the interface due to differing hardnesses and deformation behaviors of the specimens. This tended to lower the threshold deformation but did not affect the weld strength at high deformations. High deformations were therefore used for roll welding of dissimilar metal couples. Some of their data is shown in Figure 16 and Table III.

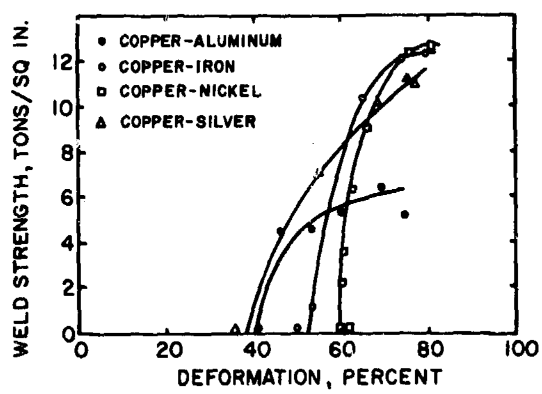


Figure 16. Weld Strength as a Function of Deformation for Various Dissimilar Metal Pairs
Rolling was conducted at room temperature[65]

Table III. Welding of Insoluble Metals[64]

System	Deformation, percent	Treatment	Weld Strength, tons/sq in.
Cd-Fe	43	Cold rolled	1.7
	59	Cold rolled	1.9
Fe-Pb	41	Cold rolled	0.4
	46	Cold rolled	0.6
	59	Cold rolled	0.8
Cu-Pb	57	Cold rolled	0.74
	57	3 hr at 290°C	0.84
Cu-Mo	54	Rolled at 600°C	4.5
	65	Rolled at 600°C	7.4
	69	Rolled at 600°C	10.0
	65	Rolled at 600°C 2 hr at 875°C	8.4
	58	Rolled at 900°C	8.0

Solid-metal shear strengths of the weaker components (tons/sq in.):

Cadmium	4.0	
Lead	0.85	
Copper	12.0	after treatment at 600° and 900°C.

Roll welding of miscible metals produced strong welds, but with subsequent heat treatment at elevated temperatures, two systems (Cu-Ni, Fe-Ni) were considerably weakened by the development of diffusional Kirkendall porosity near the interface. Immiscible cubic-metal systems welded readily, approaching the strength of the weaker metal at high deformations. With a hexagonal metal in the couple (Cd-Fe), a lower weld strength was observed as experienced in autogenous welding. Immiscible systems (Cu-Pb and Cu-Mo) displayed an increase in weld strength when subjected to postheating. Couples forming intermetallic compounds produced divergent behavior depending on the nature of the compound. Brittle intermetallic layers were detrimental to the weld when thick, but

had little influence when thin. If the compound was ductile, the weld was strong, independent of thickness, and failure occured in the weaker metal.

In a more recent paper,[82] the effects of surface contamination on roll welding of aluminum couples at room temperature were considered in greater detail. These impurities, principally oxide and water vapor, are prime factors for the inability to weld by roll welding techniques. This is proven, for example, by an experiment in which high-purity aluminum was baked out in vacuum at 932°F for 12 hours, machined in vacuum, and passed through the rolls (within 5 seconds after machining) in a vacuum of higher than 10^{-4} mm Hg. The threshold deformation was 10 percent compaired with 40 percent in earlier experiments in the atmosphere after scratch brushing. It is believed that the threshold deformation is required in order to disrupt surface contaminants and allow metallic contact. Also suggested is the possibility of reaction bonding between aluminum and oxygen (of the water molecules) at the interface with the hydrogen entering into solution in solid aluminum.

Because regions of metallic welding are restrained from yielding by the adjacent regions of bulk metal, the strength of the welded regions is believed to be greater than the base metal strength. An equation was developed in which the ultimate shear strength of the weld divided by the ultimate shear strength of the solid metal was equal to R(2-R). R was the fractional reduction in thickness. This equation was experimentally verified if the deformations were considerably above the threshold deformation or if (for aluminum) the oxide layers were thick, such as those formed in air above 900°F. Data for aluminum are given in Figure 17.

In addition to a discussion of the mode of oxide fracture and the effect of oxide layers on joint strength, its influence on the threshold deformation is important. Once the oxide layers have fractured and exposed the clean, underlying metal, the oxide probably has little influence on metallic welding unless the deformation is low or the oxide layers are thick. These latter conditions will increase the difficulty of extruding the metal through the small gaps in the oxide layers. In the case of thick intermetallic layers,[64] it was shown that the layers had to break apart to a critical separation before the base metals could be forced through the cracks and form a weld. Similar results have been observed in contact resistance experiments where a large drop in the resistance occurred at a specific deformation. This deformation increased with oxide thickness.

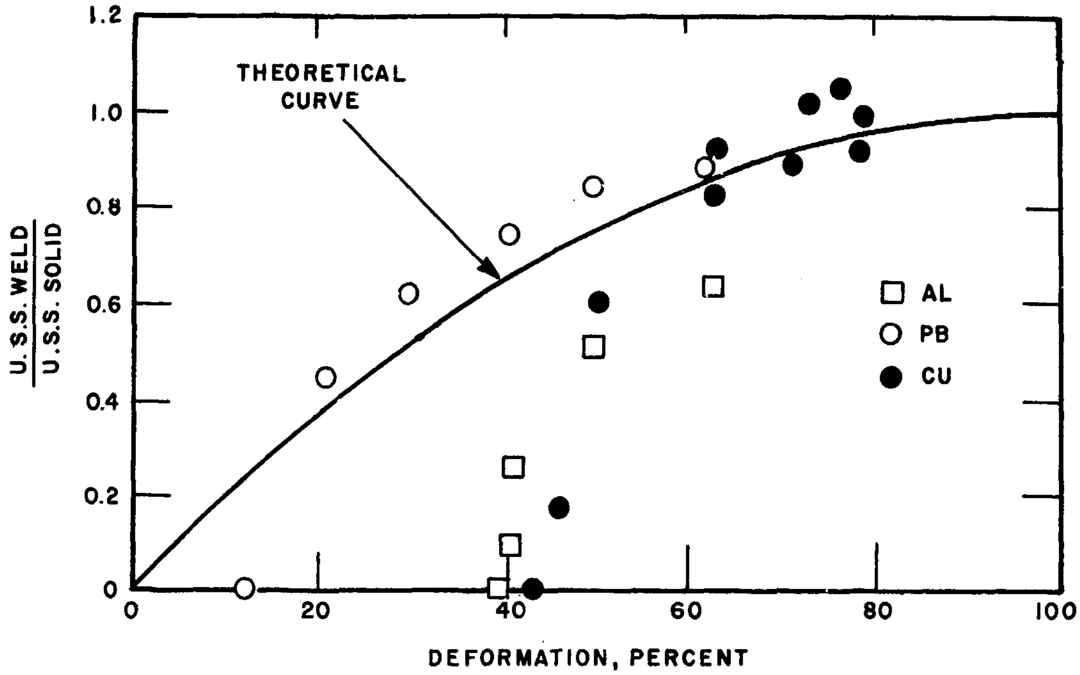


Figure 17. The Strength of Roll Welded Room-Temperature Deformation Welds[63, 84]

In a study aimed at developing low temperature roll welding techniques for flat plate composites of Type 410 stainless steel and Zircaloy 2,[86] the following conclusions were made:

1) The method employed for surface preparation was the most influential factor for lowering the threshold deformation. Of the several techniques that were investigated, degreasing followed by scratch brushing was found to be superior.

2) The amount of deformation was the parameter that most greatly influenced the joint strength.

3) The effects of intermediate material usage or surface finish on the threshold deformation were small.

4) The composite joint strength decreased with increasing deformation above the threshold deformation possibly due to a reduction in the mechanical component of the weld strength as the deformation was increased.

5) While both mechanical and metallurgical welds were formed, at high deformations (single-pass reductions greater than 70 percent) there was evidence from diffusion-zone formation that surface contact was improved and that the metallurgical welding component was augmented.

Roll welding has been used for the fabrication of titanium sandwich panels and other structural shapes.[3, 87, 88, 89, 90, 91, 92, 93, 94, 95] Truss-core panels, having the structural members supported by a matrix material of mild steel, can be fabricated by a hot plate rolling mill reduction sequence. The design configuration and assembly for truss-core panels are shown in Figures 18 and 19. A pack assembly for a panel stiffened with ribs is shown in Figure 20. The cleaned and assembled pack is sealed by welding, evacuated, and outgassed at 1600°F for about 2 hours. For most titanium alloys, the rolling temperature lies in the range of 1400° and 1800°F, and the reductions are between 60 and 90 percent of the initial pack thickness. Subsequent to rolling, the composite can be formed with conventional equipment in the same manner as a solid plate. After forming, the mild steel supporting structure is removed by leaching with a nitric acid solution.

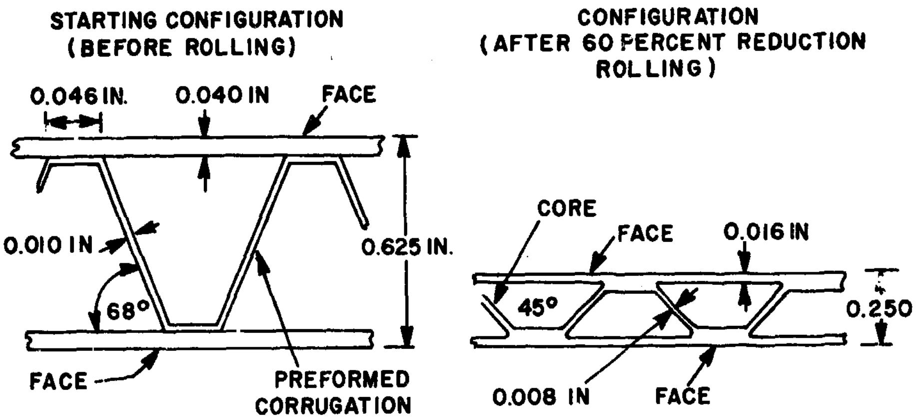


Figure 18. Design Configurations for a Truss-Core Panel[88]

Materials that have been investigated and found to be suitable for this method of fabrication are:

1) Aluminum alloys, e.g., 2024 and 5052

2) Titanium-alpha and alpha-beta alloys

3) 300-series stainless steel

4) PH 15-7 Mo stainless steel

5) Nickel-base alloys, e.g., Rene' 41 and Inconel

6) Refractory metals and alloys, e.g., columbium, tantalum, molybdenum, and tungsten.

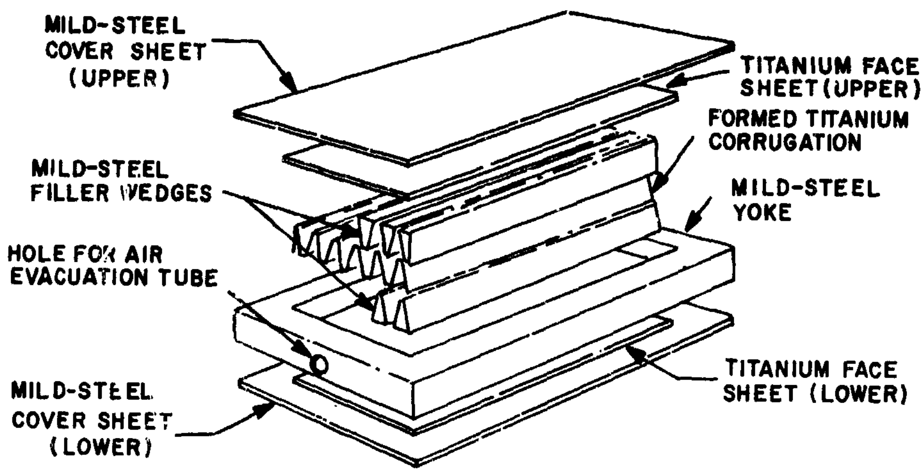


Figure 19. Pack Assembly of Corrugated Truss-Core Sandwich Panel for Roll Welding[88]

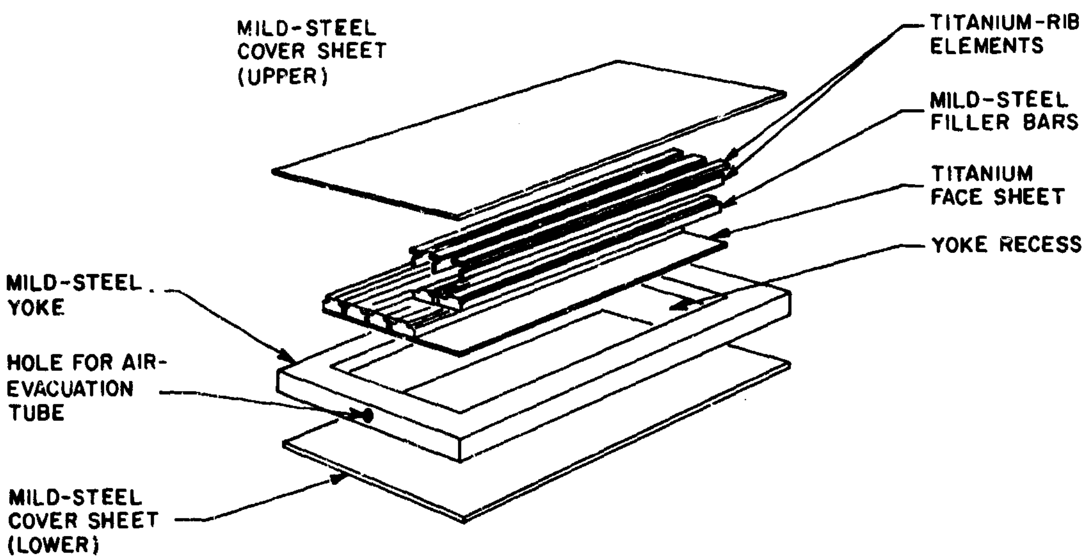


Figure 20. Pack Assembly of Structure-Stiffened Panel for Roll Welding[88]

Roll welds between Cb and Mo, B66 columbium base alloy and TZM molybdenum base alloy, Ta and W, W and Ta-10W, Cu and Ti, stainless steel and Ti, and stainless steel and Ta have recently been reported.[96] Reductions of 5 to 40 percent at 1000° to 1700°F were employed in a highly pure argon atmosphere (InFab process).

3. Joint Designs Suitable for Diffusion Welding

The design of a joint to be fabricated by diffusion welding may be based on several considerations, including base metals, welding method, and service requirements.

If the base metals to be joined are dissimilar and have unequal coefficients of expansion, it may be necessary to design the joint so that stresses are not introduced that can rupture the weld.

Availability of a particular welding method may influence the selection of a joint design. For example, any of the three diffusion welding methods could be employed to butt a solid cylinder to a flat plate. If the metals were dissimilar and the bar had a greater coefficient of thermal expansion than the plate, they could possibly be joined for the same configuration without any equipment for pressure application other than a furnace. These two possibilities are shown in Figure 21.

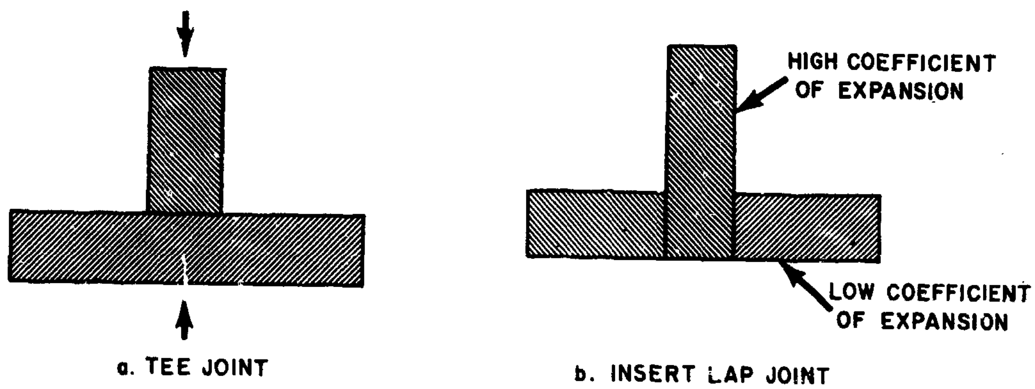


Figure 21. Joint Designs Utilizing Different Diffusion Welding Methods

Close attention to the intended service conditions must be observed. Requirements for the joint might include:

1) High tensile strength.
2) High shear strength.
3) Resistance to tearing.
4) Leak tightness.

5) Resistance to corrosion.

6) High electrical conductivity.

7) Impact resistance.

8) Resistance to fatigue.

The basic joints may be considered, as in brazing,[97] to be the butt and the lap. The lap joint and variations of it are shown in Figure 22. The butt and lap joint (f) in this figure requires pressure application from two directions.

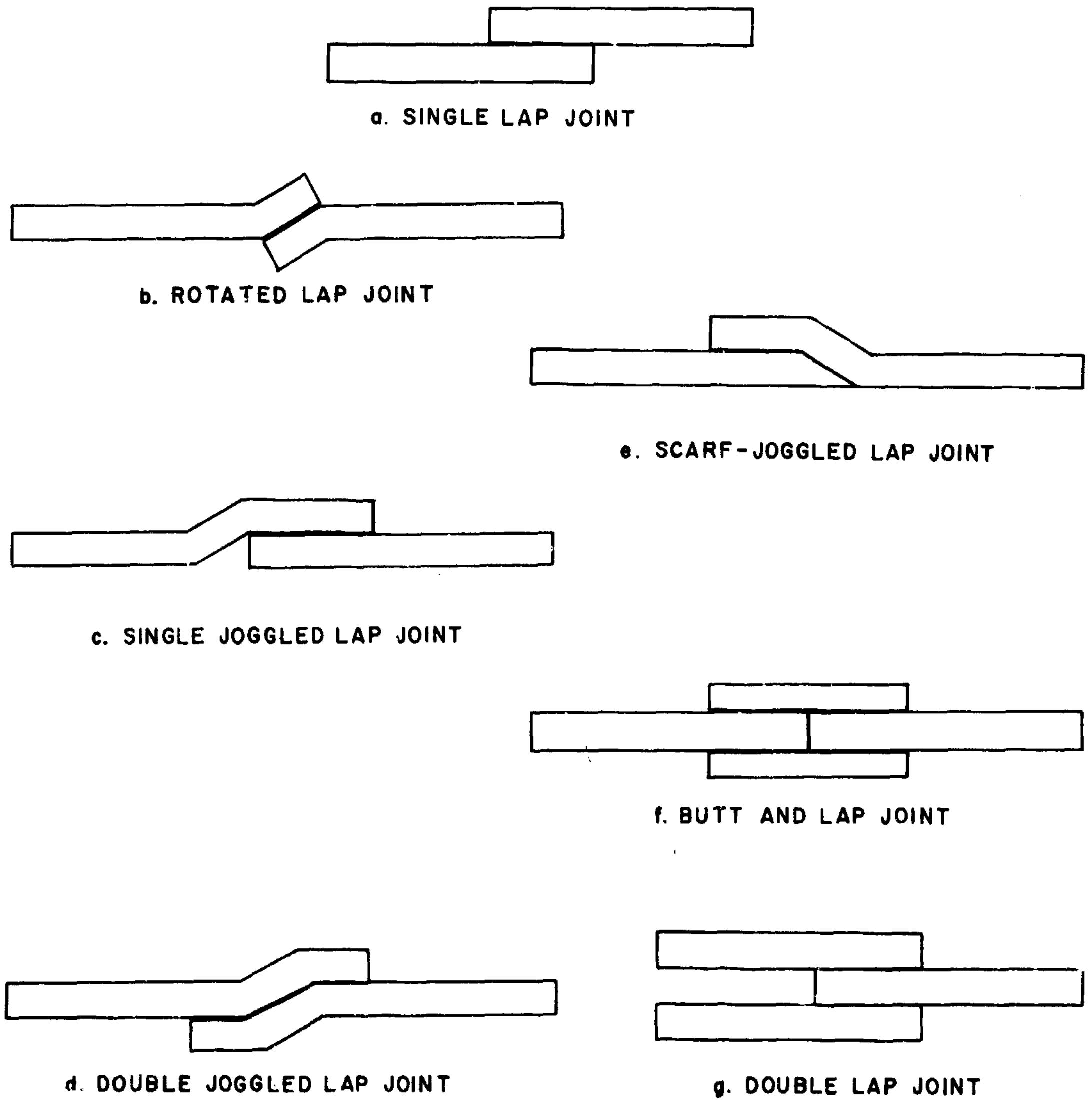


Figure 22. Variations of Lap Joints for Diffusion Welding

The tee joint, which may be considered a form of the butt joint, is shown with several variations in Figure 23. A butt joint between tubes was described earlier in the section dealing with press welding and was shown in Figure 9.

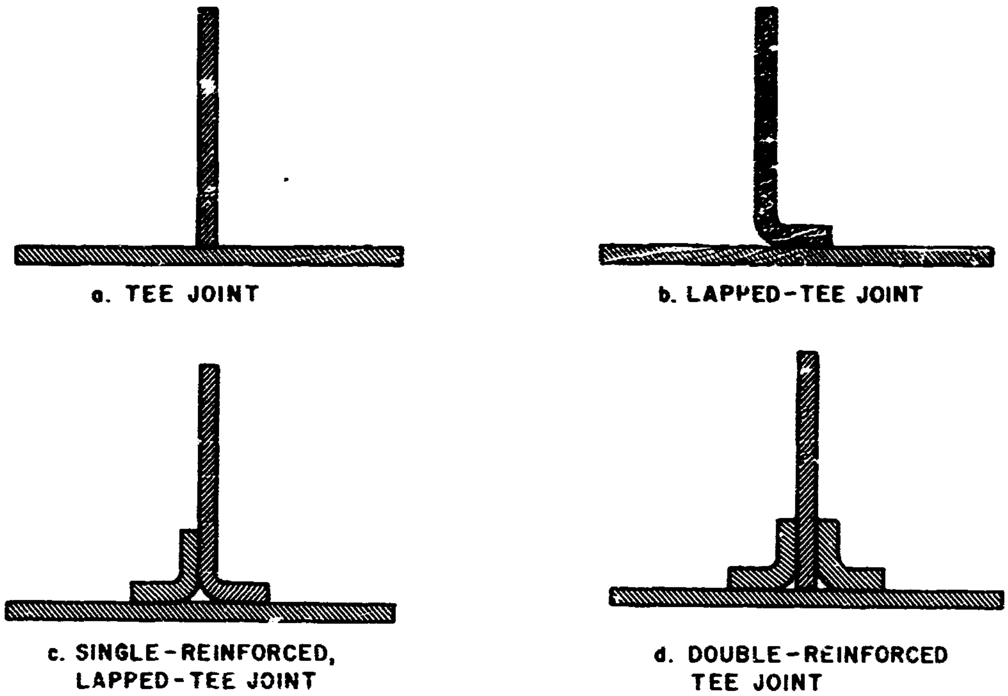


Figure 23. Variations of Tee Joints for Diffusion Welding

High strength joints can be made by increasing the area of the joint interface (increased overlap of lap joints) or reinforcing the members strengthened by increasing the joint area as shown in Figure 24 or by reinforcing as in the butt and lap joint of Figure 22(f). Impact resistance of a butt joint can be increased by increasing the joint area.

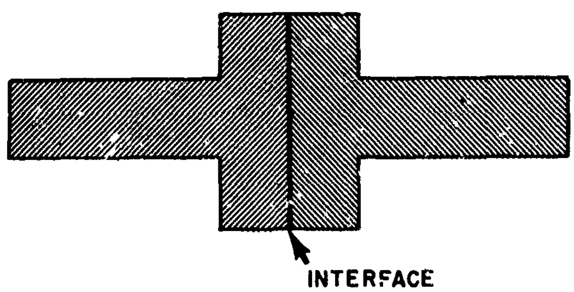


Figure 24. Butt Joint Strengthened by Increased Joint Area

Resistance to tearing of diffusion welded joints can be achieved by reducing or eliminating regions of stress concentration. Alteration of the design, so that light and heavy sections are not joined, and reinforcement are also helpful.

Leaktight diffusion welds can be obtained by proper control of the welding operation. Increased joint area reduces the electrical resistance of joints.

Fatigue life can be improved by designing to permit joint flexure and removing points of stress concentration. The double lap joint has been shown to have good fatigue resistance.[7]

Corrosion resistance can be improved when similar metals are diffusion welded by eliminating small gaps and narrow cracks. Material selection for the service environment must be carefully made. Galvanic corrosion between dissimilar metals can be reduced by careful material selection, based on the galvanic series, for example, and by enlarging the surface area of the anodic member as much as is practical.

4. Joint Designs Suitable for Roll Welding

From the earlier section dealing with roll welding, it can be seen that several joint configurations are feasible. Lap joints and clad structures are readily formed. In addition to lap joints, butt and tee joints are present in core-sandwich panels.

Designing for a specific application entails consideration of the roll welding process parameters, the materials required, and the ultimate service conditions.

Section IV. NONDESTRUCTIVE TESTING OF DIFFUSION-WELDED JOINTS

Nondestructive tests are used in a variety of applications to detect the presence of defects such as cracks or voids, variations in structure, the thicknesses of materials and coatings, and, in some instances, the strength of welded joints (both metallurgically welded and adhesive bonded). The reasons for the use of these tests are diverse, but of primary importance is the need to ensure reliability and prevent accidents. As pointed out in the Nondestructive Testing Handbook,[98] the nondestructive tests to be utilized "must be designed, specified, and scheduled for validity and reliability in each individual application." The information required to accomplish this includes such data as service loads, conditions of use, and limits of acceptability or rejectability.

The purposes of the following discussion are to point out those nondestructive testing methods that are likely to be useful in the inspection of diffusion welded joints; to summarize the reported experience in the inspection of diffusion welded joints; and to give the limitations of the present state-of-the-art in the inspection of diffusion welded joints. As pointed out above, the tests to be utilized on any particular joint are determined by the details of that joint. Such factors as shape and size, type of material, and accessibility must be considered. Therefore, the following discussion will deal primarily with general principles that should be applicable to the inspection of most diffusion welded joints.

1. Selection of Test Methods

The first step in the selection of nondestructive test methods that may be useful is to consider what types of defects may be encountered in diffusion welded joints. Generally, there are four classes of defects that might be found in diffusion welded joints:

1) Voids (cracks).
2) Unwelded areas (lack of weld).
3) Inclusions.
4) Weak welds.

The first three classes are illustrated in Figure 25. In this discussion of nondestructive testing, cracks have been included under the class of voids, since many voids in diffusion welds have large length-to-width ratios. Thus, the detection of these voids is similar to the detection of cracks. The primary distinction between a void and an unwelded area

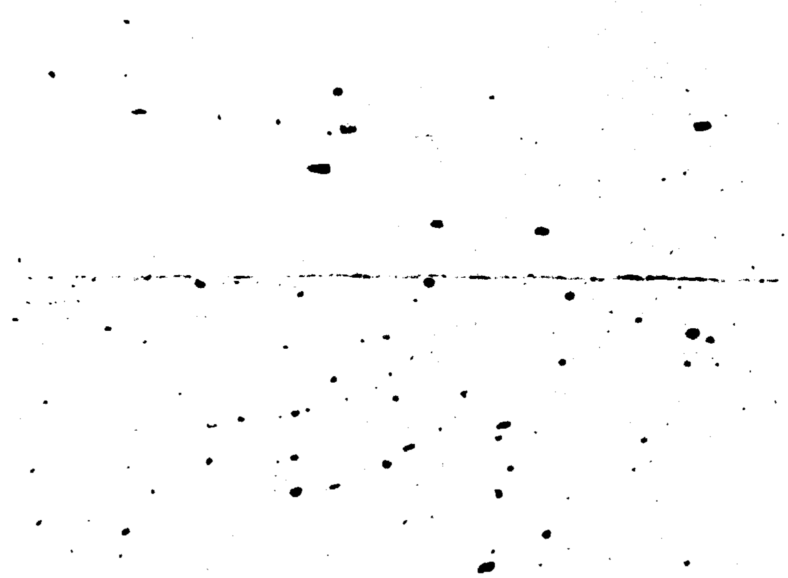
is that the void represents a definite separation across the bond line, while, at an unwelded area there is good mechanical contact but no weld. Weak welds are those in which there are no detectable defects, but if tested destructively have low strengths. Therefore, weak welds are usually found only by destructive tests. Only in a few fields, such as adhesive bonding, have similar defects been found by nondestructive tests. Presumably, weak welds result from insufficient solid state diffusion.

Assuming the above four classes of defects are those which must be detected, the applicable nondestructive tests can be selected. Table V, adapted from the Nondestructive Testing Handbook,[98] is a guide in this selection. The data in Table V are intended to be indicative, not conclusive. They are an initial guide in the selection of test methods. The table is based on the premise that any method is applied to a specific problem with adequate equipment, high grade materials, qualified personnel, and normal production-testing conditions. The various types of magnetic particle tests have been deleted from Table IV, because they are not applicable to aluminum, beryllium, and other nonmagnetic materials, which are of primary interest in this report. The defects considered in the table are those that are the same as or similar to the ones expected in diffusion welded joints.

250X Nital Etch

a. Voids in Diffusion Welded Plain-Carbon Steel

Figure 25. Defects in Diffusion Welded Joints



500X As Polished

b. Unwelded Area in Diffusion-Welded Copper

250X Picral Etch

c. Inclusions in Diffusion Welded Plain-Carbon Steel

Figure 25. (Concluded)

Ta

Defect	Penetrating-Radiation Tests**			Ultrasonic a				
				Contact-Pulse Reflection			Immers Reflec	
	Film Radio-graphy	Fluor-oscopy	Radio-isotopes	Normal Beam	Shear Wave	Surface Wave	Normal Beam	A
Minute surface cracks	U	U	U	U	U	U	U	
Internal cracks	G(F)	G(U)	G(F)	G	G	U	G	
Internal voids	G(F)	G(U)	G(F)	G	G	U	G	
Metallurgical variations	F	P(U)	F	F(P)	P(U)	U	G(F)	
Laminations (sheet and plate)	U	U	U	F(G)	F	U	G	
Shrinkage cracks (welds)	G****	G**** (F****)	G****	U(F)	F	P(F)	F	
Inclusions (welds)	G(F)	F(U)	G(U)	F(G)	F(G)	U	G	
Lack of fusion (welds)	G	F	G	U(F)	F	U	F	
Lack of weld	F	U	U	F	F	U	F	

*Code letters defined as follows: G - good; F - fair; P - poor; U - unsuitable. Letters in parentheses indicate a difference in suitability for magnetic (heavy) metals and nonmagnetic (light) metals. Letters in parentheses is for magnetic metal.

**Size of defect found depends on thickness of section.

***Defects must be open to a surface to be located with penetrants.

****Provided beam is parallel to the cracks.

Table IV. Comparison of Suitability of Nondestructive Tests

Jltrasonic and Sonic Tests						Electromagnetic Tests			
Immersion-Pulse Reflection			Through Transmission	Resonance	Natural Frequency	Eddy Current	Magnetic Field	Leakage-Field Pickup	Direct-Current Conduction
Normal Beam	Angle Beam	Surface Wave							
U	U	U	U	U	U	F	U(P)	F(U)	F
G	G	U	G	P	U	P(U)	U(P)	U	P
G	G	U	G	P	U	P(U)	U(P)	U	P
G(F)	P(U)	U	F	U	U	G	U(G)	U	P
G	F	U	G	G	U	U	U	U	P
F	F	P	P	P	U	F	U(P)	U(G)	F
G	F(G)	U	F(G)	U	U	P	U(P)	U(F)	U
F	G(F)	U	G	U	U	F	P	F	P
F	F	U	G	G	U	U	U	U	U

table.
ieavy)

destructive Tests

	Electromagnetic Tests				Liquid-Penetrant Tests***		
Natural Frequency	Eddy Current	Mag-netic Field	Leakage-Field Pickup	Direct-Current Conduction	Visible-Dye Penetrants	Fluores-cent Dye Penetrants	Filtered Particles
U	F	U(P)	F(U)	F	G(F)	G	U
U	P(U)	U(P)	U	P	U	U	U
U	P(U)	U(P)	U	P	U	U	U
U	G	U(G)	U	P	U	U	U
U	U	U	U	P	F(P)	F(P)	U(P)
U	F	U(P)	U(G)	F	G	G	U
U	P	U(P)	U(F)	U	U	U	U
U	F	P	F	P	U	U	U
U	U	U	U	U	U	U	U

3

Based on Table IV, two classes of nondestructive tests, penetrating radiation and ultrasonic tests, may be useful for the inspection of diffusion welds. Only one, ultrasonic through transmission, comes close to having a "good" capability for voids and cracks, inclusions, and lack of weld. Other ultrasonic tests and penetrating radiation tests appear less suitable. Generally, electromagnetic and liquid penetrant tests are not suitable. The applicable tests are reviewed in the following discussion.

2. Applicable Test Methods

As indicated above, two general categories of test methods, penetrating radiation and ultrasonic tests, appear suitable for the nondestructive inspection of diffusion welded joints. The literature survey on diffusion welding revealed three other specific tests, thermal, direct current conduction, and leak, which have been utilized to nondestructively inspect diffusion welded joints. With a few exceptions, all of the references to the inspection of diffusion welded joints dealt with applications in the atomic energy field. Many of these dealt with the inspection of cladding-to-core welds in fuel elements. The following is, therefore, a general discussion of the principles of the selected inspection techniques.

a. Penetrating-Radiation Tests

Penetrating-radiation tests include three general classes:

1) Film radiography.

2) Fluoroscopy and X-ray imaging.

3) Radioactive isotope radiography.

Each of these depends on differential adsorption of radiation to detect defects. The radiation penetrates through the part, and the emergent intensity is proportional to the absorption coefficient of the part. A defect, such as a void, alters the absorption coefficient of that portion of the part. The difference in intensity is detected by a suitable detector such as a film. The absorption of a part depends upon thickness, density, and atomic nature of the material. In the inspection of a part composed of two dissimilar materials or diffusion welded using an intermediate material, the differences in absorption between the different materials may cause problems. Table V gives the approximate radiographic equivalence factors of several metals. If the radiographic exposure for a given thickness of one material is known, the equivalent thickness of another material is found by using the appropriate

Table V. Approximate Radiographic Equivalence Factors* for Several Metals[98]

Metal	X-Rays								Gamma Rays			
	50 Kv	100 Kv	150 Kv	220 Kv	400 Kv	1000 Kv	2000 Kv	14-24 Me	Iridium 192	Cesium 137	Cobalt 60	Radium
Magnesium	0.6	0.6	1.05	0.08								
Aluminum	1.0	1.0	0.12	0.18					0.35	0.35	0.35	0.40
2024 Aluminum alloy	1.4	1.2	0.13	0.14					0.35	0.35	0.35	0.35
Steel		12	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Type 304 stainless steel		12	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Copper		18	1.6	1.4	1.4				1.1	1.1	1.1	1.1
Lead			14	12			5.0	2.5	4.0	3.2	2.3	2.0

*Aluminum is used as the standard metal at 50 kv and 100 kv, and the steel at the higher voltages and for gamma rays.

factor. For example, at 100 kv 12 inches of steel are equivalent to 1 inch of aluminum. This illustrates the difficulties that can be encountered in the radiography of dissimilar metal combinations.

One of the important considerations in radiographic inspection is the sensitivity of the process, i.e., the smallest flaw that can be dedetected under a given set of conditions. It has been suggested[99] that specifications for nondestructive testing divide the work into three classes in order of decreasing sensitivity requirements (Table VI).

Table VI. Sensitivity Requirements

Quality	Class of Work	Sensitivity, Minimum Perceptible Penetrameter Hole Diameter* (in.)
I	Nuclear	1 T or 0.010**
II	Pressure	2 T or 0.020**
III	Structural	4 T or 0.040**

*T = thickness.
** = Whichever is greater.

As pointed out previously, the sensitivity of penetrating radiation tests is less than optimum when the beam is not parallel to planar defects such as cracks. Furthermore, past experience indicates that the largest defects found in diffusion-welded joints have a maximum size (diameter for voids, width for cracklike defects) of about 0.010 inch. Therefore it appears that conventional penetrating-radiation tests will have limited applicability to the inspection of diffusion-welded joints. However, there are certain specialized techniques that may be useful in the inspection of thin-section (about $^1/_4$ inch maximum) components. These are microradiography and television X-ray imaging.

Microradiography has been employed to detect small voids in diffusion welded joints in copper.[28] In this technique a special fine-grained film is exposed with X-radiation and then subsequently enlarged up to about 400 times. Cunningham and Spretnak detected voids less than 0.001 inch in diameter by this method. However, the technique is not suitable for the inspection of large areas nor of thick sections.

Television X-ray imaging has undergone considerable development over the past few years.[100] The system uses small diameter, photoconductive target types of television camera tubes as direct-sensing media for penetrating radiation such as X-rays. Images from these camera tubes are transmitted as video signals, amplified, and reproduced upon the phosphor screen of large diameter television picture tubes to provide direct electronic image enlargements. Some capabilities of the inspection system are as follows:

1) Detail resolution of the order of 10 microns.

2) X-ray image enlargements of the order of 30 to 40 diameters.

3) True 2 percent, 1 T penetrameter sensitivities in steel missile case materials $^1/_8$ to $^1/_4$ inch thick.

4) Continuous scanning (in-motion viewing) of test objects without objectionable image lag or blurring.

The present systems are limited to the relatively thin materials. Furthermore, it is unlikely that this technique will detect back-of-weld defects or weak welds.

b. Ultrasonic Tests

As indicated in Table V, there are several types of ultrasonic tests. Most of these tests, however, depend on the effect a defect has on the transmission of ultrasonic waves in the part being inspected. The advantages of ultrasonic inspection, as given in the Nondestructive Testing Handbook,[98] are as follows:

1) High sensitivity, permitting the detection of minute defects.

2) Great penetrating power, allowing the examination of thick sections.

3) Accuracy in the measurement of flaw position and estimation of flaw size.

4) Fast response, permitting rapid and automated inspection.

5) Need for access to only one surface of the part.

Certain test conditions may limit the application of ultrasonic tests.[98] These include:

1) Unfavorable sample geometry, for example, size, contour, complexity, and defect orientation.

2) Undesirable internal structure, for example, grain size, inclusion content, structure porosity or fine dispersed precipitates.

The primary potential advantage of ultrasonic tests in the inspection of diffusion welded joints is that disk-like defects or cracks of almost zero thickness are readily detected. It has been reported that, in certain instances, an ultrasonic-transmission-inspection technique was capable of distinguishing between a strong metallurgical weld and a weak mechanical bond.[101]

Although the various ultrasonic tests given in Table IV differ in many respects, certain basic principles are common to all types. When an ultrasonic wave intersects a plane interface the following can occur:

1) Reflection.

2) Refraction.

3) Mode conversion.

4) Diffraction.

5) Combinations of 1 to 4.

These effects can be altered by surface roughness, specimen curvature, structure variations, irregular defect shapes, and nonuniform beam characteristics.

The nature of the material on either side of the interface is also important. This factor is very significant in the inspection of joints between dissimilar materials or diffusion welded joints utilizing one or more intermediate materials. Consider the relatively simple case of the reflection of ultrasonic plane waves normally incident on a planar interface between two materials. The percent reflection depends on the impedance mismatch between the two materials. Calculated values for the intensity of the reflected beam as a percentage of the incident beam intensity are given in Table VII.

Table VII. Calculated Values

Material Combination	Intensity of Reflected Beam, percent
Al-Al	0
Al-Steel	21
Al-Ni	24
Al-Air	~100
Steel-Ni	0.2
Steel-Air	~100

The presence of several interfaces across a narrow region further complicates the inspection problem and makes the effects of defects on the ultrasonic waves more difficult to discern.

Ultrasonic through transmission has been employed extensively in the nuclear field to inspect fuel cladding welds in fuel elements fabricated by roll welding, diffusion welding, and braze welding.[98, 101, 102, 103] In this method continuous or pulsed ultrasonic waves are sent from one transducer through the part to a second transducer. The transmission technique depends on the principle that certain specific changes in the sample will produce significant changes in the ultrasonic beam passing through it. The defect detectability of this type of system depends primarily on the ratio of defect area to beam size and the separation between the defect and the transducers.[98] Beck[101] reported that such a system was capable of discriminating between metallurgical welds and mechanical bonds. Beck's system was capable of detecting unwelded areas of $^1/_{32}$ inch diameter in clad fuel elements. The same system detected microporosity, microinclusions, and other defects in cast uranium. Ross and Leep[103] reported that their through-transmission system could detect unwelded areas as small as $^1/_8$ inch in diameter in aluminum-clad uranium fuel elements. Their system could function through 8 inches of uranium and, therefore, probably through several feet of aluminum or steel.

Although the through-transmission technique has been found most suitable in past studies of the inspection of welds, some of the other ultrasonic tests such as contact or immersion pulse reflection could be used in certain instances. These methods utilize the energy reflected from a defect to detect and locate it. The pulse reflection methods are best for precisely locating flaws;[102] furthermore, they require access from only one side of the part. Some values reported for minimum size of defect detectable are as follows:

1) Crack 0.025 inch long at a depth of 1 $^1/_4$ inch in steel and 0.045 inch long cracks in aluminum.[104]

2) Nonmetallic inclusions 0.004 to 0.100 inch long in a steel forging.[105]

3) Unwelded areas smaller than $^1/_{32}$ inch diameter; between $^1/_4$ inch thick stainless steel and 2 inch thick electroformed copper.[106]

Immersion pulse reflection testing has been applied to the inspection of face sheet-to-core diffusion welds in titanium alloy,[107] refractory metal,[6] and superalloy[6] honeycomb sandwich. Reportedly, satisfactory results were obtained with the normal ultrasonic inspection procedures employed on brazed sandwich.

Most of the above reports indicated that the equipment and procedures were developed especially for the particular problem. Similar development will be required to utilize ultrasonic techniques to inspect diffusion welded components; however, ultrasonic tests appear more promising for this task than any of the other inspection methods.

c. Other Tests

In certain specific applications, particularly in the nuclear field, other specialized nondestructive tests have been utilized to inspect diffusion, roll, or brazed welded joints. These included direct current conduction, thermal, and leak tests.

A modified direct current conduction method was developed to inspect core-to-clad welds in flat plate fuel elements.[108] The technique measured the change in electrical resistance resulting from the presence of an unwelded area, voids, or oxide inclusions. The system was capable of locating unwelded areas at least as small as $^1/_{16}$ inch in diameter and possibly as small as $^1/_{32}$ inch in diameter.

Thermal tests have been used extensively to inspect fuel element core-to-clad welds,[102] brazed welded, and adhesive bonded honeycomb.[110, 119] Just as the direct current tests utilize the effect of defects on electrical conductivity, the thermal tests utilize their effect on thermal conductivity. Thermal tests employ temperature sensitive coatings or temperature measuring devices such as infrared sensors to measure temperature differences resulting from variations in thermal conductivity. Defects such as unwelded areas markedly reduce the thermal conductivity of local regions in a welded assembly. This type of test has many disadvantages such as (1) relative slowness, (2) sensitivity to ambient temperature conditions, and (3) difficulties of precise control of heat input and material temperature measurements.[98] Few cases exist in which other tests are not more sensitive and provide more useful results with less difficulty.[98]

Leak tests are useful in the inspection of joints in piping, pressure vessels, and other containers. Usually a gas is used as the probing media. Generally, the component under test is pressurized or evacuated and a suitable detector used to locate leaks. Helium is commonly used in conjunction with a special mass spectrometer or "leak detector". Commercially available instruments can detect leaks as small as 2×10^{-11} std cc/sec. Leak tests have been used in laboratory studies to inspect aluminum-to-stainless steel diffusion welded joints in tubing[76] and stainless steel-to-stainless steel joints in piping connectors.[111] In the latter study, good correlation was found between joint strength and leaktightness. Once satisfactory welding conditions had been established, strong joints were also leaktight.

Two important factors in nondestructive testing should be emphasized. First, a nondestructive test must be selected (or designed) and evaluated for validity and reliability in each individual application. Second, the limits of acceptability or rejectability must then be established using appropriate destructive tests in conjunction with the selected nondestructive method. Very little quantitative information is presently available on the effects of various types, sizes, and numbers of defects on the properties of diffusion welded joints.

Based on the available data, ultrasonic tests appear most suitable for the inspection of diffusion welded joints; however, other methods may be useful in certain applications.

Section V. DIFFUSION WELDING OF BERYLLIUM, ALUMINUM ALLOYS, AND STAINLESS STEELS

Similar and dissimilar joining of beryllium, aluminum, and stainless steel requires the consideration of the base material properties, the design of the hardware, intended service environment, and processing method employed for the six possible combinations, which are:

1) Be-Be.
2) Al-Al.
3) stainless steel-stainless steel
4) Be-Al.
5) Be-stainless steel.
6) Al-stainless steel.

A summary of diffusion welding studies for five of these combinations is presented at the end of this section. The literature survey did not reveal information concerning the diffusion welding of Be to Al.

For these metal combinations, cleaning procedures and ranges of welding parameters (temperature, pressure, time) are presented. For the material combinations in question, there are a number of different combinations of parameters that can produce sound joints. Therefore, ranges are presented rather than specific values. In most cases, the information summarized here can be used only as a guide for a more detailed development of the joining process for a particular application. By optimization of the selected joining technique and utilization of proper intermediate materials, the values of the temperature, pressure, and time of welding could probably be reduced.

The materials themselves present individual problems which must be considered in any joining operation. The mechanical and physical properties of the metals and alloys (e.g., strength, melting points, and thermal expansion coefficients) must be considered when designing a structure or developing the method for joining. Chemical properties (e.g., alloying, corrosion, and oxidation behavior) must also be taken into account. Al and Be react readily with oxygen to form surface oxides that interfere with diffusion welding. Welding in a protective atmosphere or vacuum is, therefore, recommended for these metals. It is possible, however, to diffusion weld Al in air as will be seen in the summary tables.

The selection of a cleaning procedure is dependent on the materials and joining method for the ultimate hardware being fabricated. The size and shape of the components, the speed of the fabrication sequence desired, and the surface condition desired are factors that contribute to this selection.

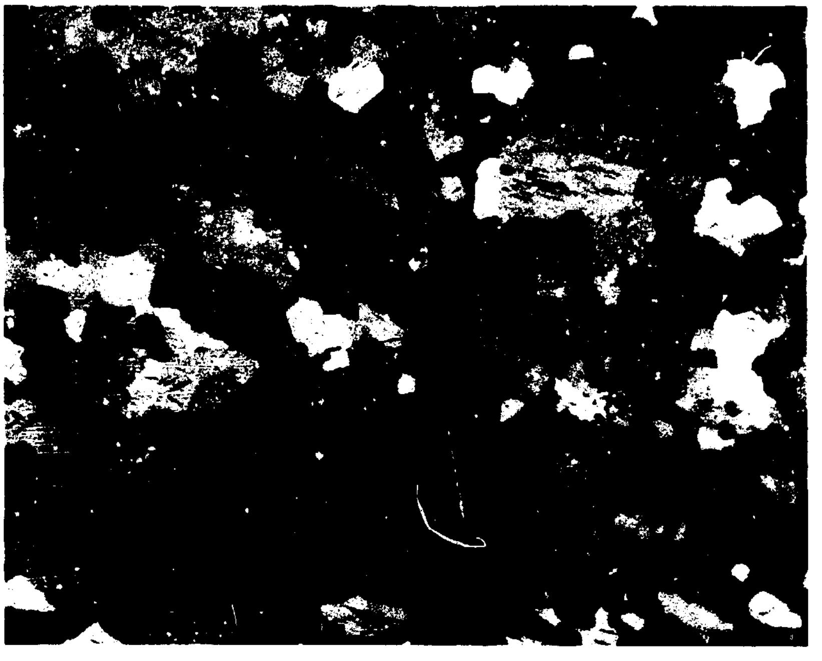
Table VIII presents the ranges of welding parameters that could be employed for diffusion welding.

Representative diffusion welded joints and structures of beryllium, aluminum, and stainless steel are shown in Figures 26 through 34.

Table VIII. Welding Parameters for Diffusion Welding Combinations of Beryllium, Aluminum, and Stainless Steel*

Materials	Welding Temp, °F	Welding Pressure, psi	Welding Time, hr
Be-Be	1500-2000	5,000-10,000	1-5
Al-Al	700-1000	3,000- 7,000	0.25-3
S.S.-S.S.	1500-2100	5,000-10,000	0.25-3
Be-Al	(850-1050)	(5,000-10,000)	(0.5 -5)
Be-S.S.	1500-2000	5,000-10,000	1-5
Al-S.S.	700-1050	5,000-10,000	0.25-5

*Parameters for diffusion welding Be and Al are derived from the data given for the joining of these metals to themselves.



250 X Polarized Light

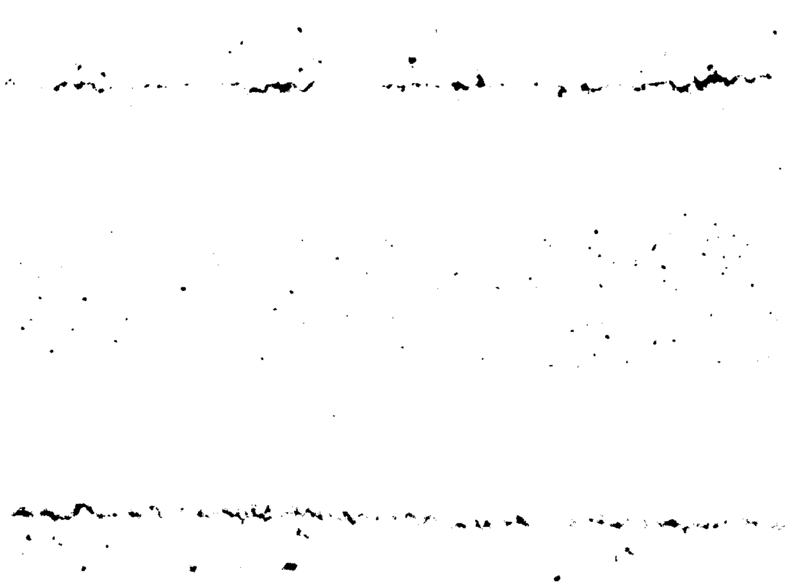
Figure 26. As-Polished Beryllium Specimen Joined By Gas-Pressure Bonding
Specimen was prepared by grit blasting and HNO_3 cleaning. Welding was carried out at 1650°F for 4 hours and 10,000 psi. Grain growth has occurred over 30 to 50 percent of the interface.

1 X

Figure 27. I-Beam Structural Member Produced from Beryllium Sheet by Gas-Pressure Bonding

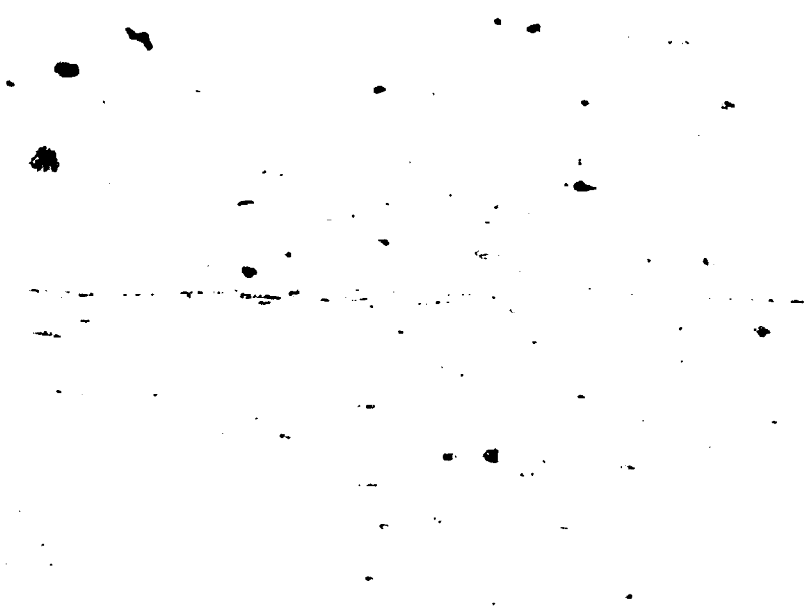
1 X

Figure 28. Truss-Supported Airframe Structure Made from Beryllium Sheet



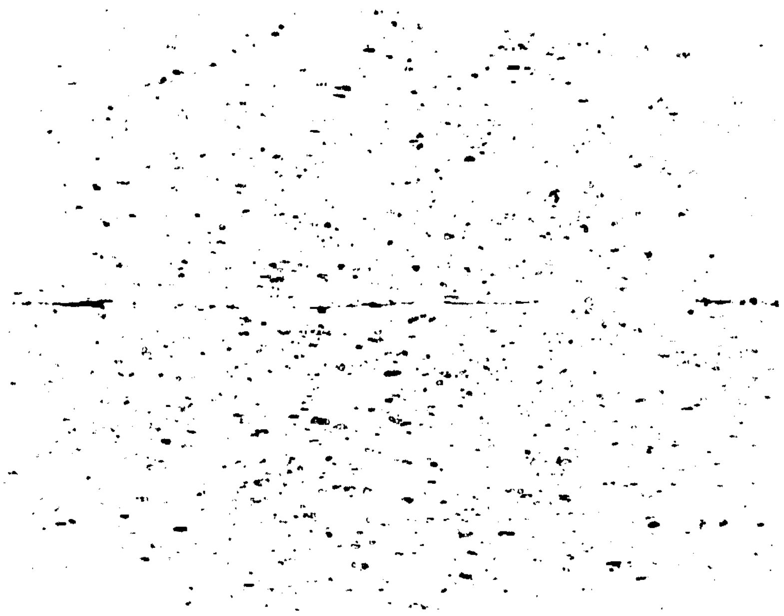
500 X Keller's Etch

Figure 29. Diffusion Welded 1170 Aluminum Using a 0.005-Inch-Thick Foil of 72 Weight Percent Ag-28 Weight Percent Cu
Welded at 1400 psi and 900°F for $^1/_4$ hour.



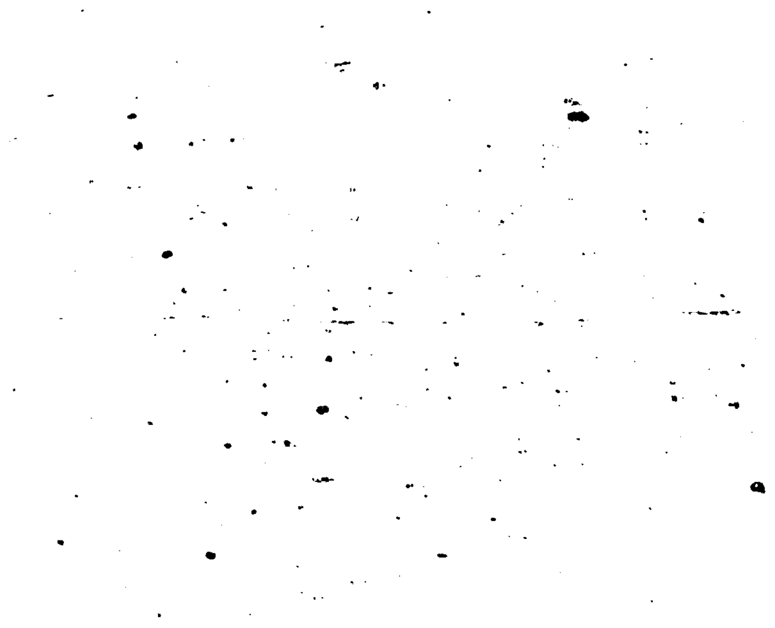
750 X　　　　　　10% NaOH

Figure 30. Diffusion Welded 1170 Aluminum Welded at 1400 psi and 900°F for 1/4 Hour



250 X 10 Percent NaOH

Figure 31. Diffusion Welded 1100 Aluminum Welded at 2200 psi and 900°F for 1 Hour



250 X 10 Percent NaOH

Figure 32. Diffusion Welded 1170 Aluminum Welded at 2200 psi and 900°F for 1 Hour

250 X ½ Percent HF

Figure 33. Diffusion Welded 7075 Alclad Aluminum Tee Section Welded at 11,000 psi in a Press and 750°F for 5-½ Hours
Tensile strength was 5300 psi.

347 Stainless

Ni Strike, 0.0005-Inch Au Plate

0.008-Inch Cu Foil

347 Stainless

100 X As Polished

Cu Foil

Au-Plate
Ni-Strike

347 Stainless

750 X As Polished

Figure 34. Diffusion Welded Type 347 Stainless Steel with Au-Cu-Au Intermediate System
Welded at 25,000 psi and 700°F for $\frac{1}{4}$ hour.
Tensile strength was 24,500 psi.

1. Surface Preparation Methods

For diffusion welding it is important that clean surfaces be brought into contact. The method used for surface preparation must therefore be capable of removing unwanted surface contaminants. Depending on the material being welded and the welding method, some roughness and cold working of the surface may also be desirable. During the development of diffusion welding processes, suitable methods for surface preparation have been established. These are given for beryllium, aluminum, and stainless steel.

a. Beryllium

Of the several techniques used to prepare beryllium for diffusion welding, one consisting of grit blasting with chilled iron grit followed by washing the surfaces in dilute nitric acid to remove embedded grit is adequate. It has been shown to be superior to a number of other methods when used prior to diffusion welding by the gas pressure bonding process.[112] Smooth, mechanically prepared surfaces were found to recrystallize near 1300°F before the welding temperature and full welding pressure were attained. The presence of numerous asperities, formed by grit blasting, permits additional deformation and recrystallization during the welding process resulting in 20 to 50 percent grain growth across the interface.

Another reference[45] has suggested that metallographically polished surfaces are required in order to provide intimate surface contact. In this work, however, the welding pressure was applied by differential thermal expansion of the specimen and a fixture. The pressures applied were probably low because intimate contact was not achieved between roughened surfaces unless temperatures above 1800°F were employed. At 1922°F, ground or polished surfaces welded to the same strengths, that is, surface roughness did not influence weld strength.

b. Aluminum

A suitable surface preparation technique for aluminum alloys would consist of:

1) Degreasing.

2) Wire brushing or other mechanical cleaning and surface working procedure.

3) Alkaline cleaning (e.g., 350 g NaOH in 2 gallons H_2O for several minutes at 70°F).

4) Acid pickling (e.g., 50 volume percent, aqueous solution of HNO_3 for about 1 minute at 70°F).

5) Rinsing in deionized water.

6) Drying.

c. Stainless Steel

Cleaning stainless steel prior to diffusion welding could be accomplished using the following procedure:

1) Degrease.

2) Pickle in 15 weight percent HNO_3-1.5 weight percent HF-83.5 weight percent H_2O.

3) Rinse in deionized water.

4) Dry.

A more complex procedure that has been successfully used prior to gas pressure bonding of 347 stainless steel[113] consisted of mechanically preparing the pieces, degreasing in alcohol, pickling in 10 volume percent HNO_3-2 volume percent HF aqueous solution for 2 minutes at 120° to 140°F, and rinsing in cold running water. Following these steps, a washing and cleaning cycle was conducted as follows:

1) Placed in trichlorethylene vapor bath (5 minutes).

2) Scrubbed in methyl ethyl ketone.

3) Scrubbed in 200-proof alcohol.

4) Placed in 200-proof ultrasonic bath (5 minutes).

5) Scrubbed in a hot Alcanox solution (180°F).

6) Rinsed in cold running water.

7) Placed in 200-proof ultrasonic bath (5 minutes).

8) Rinsed in cold running water.

9) Rinsed in hot water (180°F).

10) Blown dry with filtered air.

Selection of a degreasing chemical depends on the condition of the surfaces. Common degreasing agents include acetone, alcohol, methyl ethyl ketone, trichloroethylene liquid or vapor, and perchloroethylene liquid or vapor. Any of these should be suitable. The subject of metal cleaning is extensively discussed in Reference 114.

Final drying of metal parts can be accomplished in a number of ways. These include:

1) Blasting with a clean, dry gas (e.g., filtered air or nitrogen).

2) Swabbing with a volatile solvent such as alcohol.

3) Immersing in a hot vapor such as trichloroethylene or perchloroethylene and subsequently allowing the pieces to dry in air.

4) Placing in a heated oven.

If the welding is to be conducted in vacuum, drying can be readily accomplished immediately prior to bringing the faying surfaces into contact. Volatile surface impurities will be desorbed in vacuum at an elevated temperature.

2. Summary of Diffusion Welding Data

The following tables (IX-XIII) summarize the survey on diffusion welding of beryllium, high strength aluminum alloys, and Type 321 stainless steel. Similar and dissimilar combinations of these are included except for diffusion welding of Be to Al, for which no data were found. The tables that present data for high strength aluminum alloys and Type 321 stainless steel have been expanded to include alloys other than those specified. This was done because other aluminum and iron base alloys have welding parameter requirements similar to the requirements for the alloys specifically requested. Types 321 and 347 stainless steels, for example, are very similar, the former being stabilized with titanium additions and the latter with columbium and tantalum. Their chemical, mechanical, and physical properties are nearly the same. From the tables, it can be seen that more information was available on diffusion welding Type 347 stainless steel than any other grade.

Table IX. Summary of D

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F
Be	Plate laminate	Etch in 10 percent H_2SO_4 at 110 F	None	GPB	1650
Be	Plate laminate	Chilled-iron grit blast, diluted HNO_3 rinse	None	GPB	1650
Hot-pressed Be	Butted rods	Ditto	None	GPB	1650
Be	--	--	None	--	2010
Be	--	--	None	--	2100
Be	--	--	None	--	2190
Be	Butt rods				1560
QMV Be from -200-mesh powder	Butt 1/2-in. rods, hot pressed and extruded	Ground on SiC and polished with alumina	None	DTE in Mo screw clamp	1780 optimum

Summary of Diffusion Welding Data for Joining Beryllium

ng *	Welding Temp, °F	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Com
	1650	10,000 (gas)	4	Evacuated container	Not tested	--	2, 113	No gr acro
	1650	10,000 (gas)	4	Ditto	Not tested	--	2, 113	Grain acro perc inte
	1650	10,000 (gas)	4	"	36,400 avg (standard tensile bars)	Away from interface	2, 113, 116	>30 p grov inte failu fron inte
	2010	--	24	--	--	--	117	No gr disp inclu
	2100	--	24	--	--	--	117	Some and sion inclu
	2190	--	2-1/2	--	28,000	--	117	Comp grov inte perc effic
	1560	10,000			40,000		118, 119	Some rods
Mo	1780 optimum	--	1	Vacuum 5×10^{-5} torr	57,000 max (standard 0.226 in. diam bars)	Rough poly-crystal-line fracture surface	46	0.4 to incr inte due defo

; Data for Joining Beryllium

Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
4	Evacuated container	Not tested	--	2, 113	No grain growth across interface
4	Ditto	Not tested	--	2, 113	Grain growth across 30 to 50 percent of interface
4	"	36, 400 avg (standard tensile bars)	Away from interface	2, 113, 116	> 30 percent grain growth across interface when failure was away from original interface
24	--	--	--	117	No grain growth or dispersion of inclusions
24	--	--	--	117	Some grain growth and good dispersion of inclusions
2-1/2	--	28, 000	--	117	Complete grain growth across interface, 100 percent joint efficiency
		40, 000		118, 119	Some upsetting of rods
1	Vacuum 5×10^{-5} torr	57, 000 max (standard 0. 226 in. diam bars)	Rough polycrystalline fracture surface	46	0. 4 to 5. 6 percent increase in interface diam due to plastic deformation

elding nosphere	Weld Strength, psi	Type of Failure	Reference	Comments
uum x 10^{-5} rr	41,000 max	Rough poly-crystal-line fracture surface	46	0.1 to 0.6 percent increase in diam due to plastic deformation
itto	37,000 max	Ditto	46	Ditto
cuated ild- eel ns $^{-2}$ torr	--	--	120	--
uum			76	He minimized vaporization of Be and/or base-metal impurities
tto				
e e				
cuated ntainer	36,400 Avg	In parent metal; specimens ground to tensile bars	121	
ium	40,000-45,000		8	

Table IX. (Continued)

rface paration	Intermediate	Welding Method*	Welding Temp, °F	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strengtl psi
nd on SiC polished ı alumina	None	DTE in Mo screw clamp	1730 optimum	--	1	Vacuum 5×10^{-5} torr	41,000 m
ıd on grit ɔide ɜr	None	Ditto	1930 optimum	--	1	Ditto	37,000 m
	None	GPB	1500	10,000 (gas)	2	Evacuated mild-steel cans 10^{-2} torr	--
-	None	Mo fixture for DTE	2012		2	Vacuum	
	Ditto	Ditto	2012		4	Ditto	
	"	"	2102		2	"	
	"	"	2102		4	"	
	"	"	2012		3	He	
	"	"	2102		2	He	
with ed Fe rinse luted ₃, etch ₂SO₄, wash	None	GPB	1550-1850	10,000 (gas)	4	Evacuated container	36,400 Av
h and ched	None		1650		1	Vacuum	40,000-45,000

Table

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F
	Butt 1/2-in. rods, hot pressed	Ground on SiC and polished with alumina	None	DTE in Mo screw clamp	1730 optimum
	Ditto	Ground on 180 grit carbide paper	None	Ditto	1930 optimum
QMV Be powder; 98.5 wt % Be 1.79 wt % BeO to itself and to Type 316 S.S.	Powder outside a 0.500-in. S. S. tube. Center of tube filled with mild-steel mandrel. 1.225 overall OD x 4 in. long		None	GPB	1500
Be hot pressed	--	--	None	Mo fixture for DTE	2012
			Ditto	Ditto	2012
			"	"	2102
			"	"	2102
			"	"	2012
			"	"	2102
Be hot-pressed block	Butted rods	Blast with chilled Fe grit, rinse in diluted HNO_3, etch in H_2SO_4, and wash	None	GPB	1550-1850
Be		Smooth and unetched	None		1650

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Weldin Temp °F
Be				GPB	1650
Be	Butt rods	180-grit SiC or polish with levigated alumina		DTE Mo clamps	1500-2
Be			None	DTE Mo fixture	1500-2
Be	Be tube and solid Be plug inside tube		None	DTE Mo fixture	

*GPB - gas-pressure bonding: DTE - differential thermal expansion.

Table IX. (Concluded)

ng *	Welding Temp, °F	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comm
	1650	10,000 (gas)	4	Evacuated container			2, 79	
o s	1500-2300		1	Vacuum 5×10^{-5} torr	100 percent joint efficiency		3, 50	
o e	1500-2300		1	Vacuum 5×10^{-1} torr			49	No evid grain ing
o e								Joint wa cent s

ion.

2

, (Concluded)

...lding ...ssure, ...si	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
...0 (gas)	4	Evacuated container			2, 79	
	1	Vacuum 5×10^{-5} torr	100 percent joint efficiency		3, 50	
	1	Vacuum 5×10^{-1} torr			49	No evidence of grain coarsening Joint was 75 percent sound

3

Table X. Summary of Diffusion Weld

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F	Welding Pressure, psi
7075-T6 6061-T6 2219-T6	Single lap 1 in. x in. x 0.1 in.	Final wire brushing	None	25-ton press, quartz lamp radiant heat	450	--
7075-T6	Ditto	--	Sn, vapor deposited	Ditto	500	16,000
7075-T6	"	--	Ag electroplate	"	450	16,000
7075-T6	"	--	Ditto	"	300	16,000
6061-T6	"	--	Zn electroplate	"	450	20,000
Al clad 7075 (7072 cladding)	"	Caustic, acid, and wire brush	None	"	325	24,000
Ditto	"	Ditto	Ditto	"	325	24,000
"	"	Transverse serrations, 0.030 in. pitch x 0.025 in. deep	"	"	325	24,000
7075-T6		Vapor degr., caustic etch acid dip sand blast	Al, plasma sprayed after surface preparation	"	425	24,000
7075-T6 to Al clad 7075		Ditto	Al, plasma sprayed on 7075 interface	"	425	24,000
Al	Butted rods	Abraded just prior to bonding	Ni disk	Press, induction heating, tapered sleeve	1932	40,000
Al	Ditto	Ditto	Ditto	Ditto	1932	22,000
1100 Al to 6061 Al	1100: tube 6061: end plug	(1) Trichloroethylene (2) Oakite "160" (3) Rinse (4) HCl (6061 only) (5) Rinse (6) 7N HNO_3 (7) Rinse (8) Dry	None	GPB	1000	7,000 (gas)
Al	Disks	Several	None	GPB	950	10,000 (gas)

ary of Diffusion Welding Data for Joining Aluminum

lding emp, F	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
150	--	--	Air	<3,500		122	
500	16,000	2	Ditto	300		122	
150	16,000	4	"	6,700		122	
300	16,000	4	"	3,600		122	
150	20,000	4	"	450		122	
325	24,000	1	"	9,000-10,000		122	Optimum parameters; no deformation
325	24,000	1	"	15,000		122	Reheat treated to T6
325	24,000	1	"	26,000		122	
25	24,000	4	"	7,400 11,300 11,000		112	Reheat treated to T6; longitudinal serrations
25	24,000	4	"			112	Satisfactory weld produced
32	40,000	1/15	Vacuum 5×10^{-3} torr	20,000		16	Pressure is effectively hydrostatic. These parameters gave maximum strength
32	22,000	1/15	Ditto	6,000		16	
00	7,000 (gas)	1/3	Evacuated container	--	--	123	He-leaktight joints
50	10,000 (gas)	3	Evacuated container			124	Shaper machining to 35 μ in. rms or polishing are best. Bond-line contamination ($Al_2 0_3$) occurred with no grain growth across interface

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n

Weld trength, psi	Type of Failure	Reference	Comments
<3,500		122	
300		122	
6,700		122	
3,600		122	
450		122	
9,000-10,000		122	Optimum parameters; no deformation
15,000		122	Reheat treated to T6
26,000		122	
7,400 1,300 1,000		112	Reheat treated to T6; longitudinal serrations
		112	Satisfactory weld produced
0,000		16	Pressure is effectively hydrostatic. These parameters gave maximum strength
6,000		16	
--	--	123	He-leaktight joints
		124	Shaper machining to 35 μ in. rms or polishing are best. Bond-line contamination (Al_2O_3) occurred with no grain growth across interface

3

Table

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F	1
Al	Disks		0.001-in.-thick coatings of Ag, Cu, Au, Sn, Zn electroplated	GPB	950	1(
Al	Disks		Al, Li hydride	GPB	950	10
6061 Al				GPB	850	10.
Al			None	GPB	700	6,
Al			None	GPB	950	10,

*GPB - gas-pressure bonding
DTE - differential thermal expansion.

Table X. (Concluded)

e	Welding Method *	Welding Temp, °F	Welding Pressure psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference
ick Ag, Zn d	GPB	950	10, 000 (gas)	3	Evacuated container			124
de	GPB	950	10, 000 (gas)	3	Evacuated container			124
	GPB	850	10, 000 (gas)	1	Evacuated container			79
	GPB	700	6, 000 (gas)	1/12	Evacuated container			
	GPB	950	10, 000 (gas)	1/12	Evacuated container			62

2

ncluded)

ig e	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
gas)	3	Evacuated container			124	Stronger welds when only one disk plated due to decreased total amount of coating; not surface effect. No high hardness. Ag, Sn, and Au were best
gas)	3	Evacuated container			124	Hydride did not promote a continuous weld. Intermittant porosity and second phase appeared; weld zone was of high hardness
gas)	1	Evacuated container			79	
as)	1/12	Evacuated container				
as)	1/12	Evacuated container			62	

3

Table XI. Summary of Diffusion Welding

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F	Welding Pressure, psi
347 S.S.	Butted		Au-plated surfaces, Cu foil between 0.0005-in. Au plate and 0.008-in. Cu foil	Press, in tapered liner	500	30,000
					700	25,000
347 S.S.	--	--	None	GPB	1830	12,000
					2190	2,000
					2190	12,000
S.S.	--	--	None	GPB or press	2100	10,000
321 S.S.	Honeycomb panel	--	--	--	2200	6
347 S.S. 304 S.S.		Mechanically clean, alcohol rinse, HNO_3-HF pickle, water rinse, wash	None	GPB	2100	300-500 til 2100 F reached, then 10,000
304, 316, 347, 410 S.S., 304L		Pickled, as-rolled, belt abraded, or milled	None	GPB	2100 approx	10,000 (gas)
347 S.S.	Single lap 1/16 x 3/8 x 2 in. Overlap = 1.5 t = 3/32 in.		Ni-3 wt % Be 0.0004-in. foil	Static weight	2110	Nil. 3/8 in. diam by 1/2-in. stainless steel weight
347 S.S.	Double lap 1/8 x 3/8 x 2 in. Overlap = 1.5 t = 3/16 in.		Ni-20 Cr.-0.3 Mn-3 Be (wt %) 0.004-in. thick foil	Ditto	2080 and 2100	Ditto

 GPB - gas-pressure bonding.

ı Welding Data for Joining Stainless Steel

lding sure, ›si	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
)0	1	Air	36, 900 avg		112	Specimens were leaktight; no welds produced at 300°F
)0	0.25	Air	24, 500 avg			
)0	1/3	Evacuated container			125	Minimum welding conditions
)0	1/3					
)0	1/12				11	
)0	1-3	--	--	--	73	General conditions for diffusion welding
6	1/20	--	--	--	126	
00 til) F :hed,)00	2	Evacuated container			114, 127	Slight grain growth and excellent welding when examined metallographically
0 (gas)	1-3	Ditto	100 percent efficiency		2, 62	
·/8 in. ı by ·in. ıless l ;ht	1/12	Vacuum 7×10^{-5} torr	39, 600	Through weld in shear	15	
:to	1/6	Ditto	17, 500 avg at 1500°F	In parent metal	15	Weld strength was 20, 000 psi at 2100°F welding temperature and 15, 000 psi at 2080°F

2

Table XII. Summary of Diffusion Welc

Alloy and Condition	Specimen Geometry	Surface Preparation	Intermediate	Welding Method*	Welding Temp, °F
Be to 316 S. S.	Wafer laminate, metallographic specimen, 1/2-in. diam by 1/16 in.	Metallographic to 0.5-micron diamond abrasive and then Linde B alumina	None	Threaded plug, 60 ft-lb torque	1800
Be to 316 S. S.	Ditto	Ditto	0.002-in. Cu foil	Ditto	1500
Be to 316 S. S.	Stainless tube (0.5-in. OD by 0.049-in. wall) inside a Be tube (1-1/4-in. OD)	Stainless was Ag plated and was brushed. Be was etched, Ag plated and reamed	Approx 0.0005-in. Ag on stainless OD and Be ID	DTE	1450

*DTE - differential thermal expansion.

sion Welding Data for Joining Beryllium to Stainless Steel

ding mp, F	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
00	--	4	Vacuum 10^{-5} torr	--	--	128	Measurable interme zone; multiple fra tures appeared at interface that had high hardness
00	--	4	Ditto	--	--	128	Welded; Cu failed to diffusion of Be int failure occurred i
50	--	2	Vacuum 7 x 10^{-5} torr	--	--	129	Fine partition line p ally observed at 3 magnifications

2

r Joining Beryllium to Stainless Steel

Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
4	Vacuum 10^{-5} torr	--	--	128	Measurable intermetallic zone; multiple fractures appeared at interface that had high hardness
4	Ditto	--	--	128	Welded; Cu failed to retard diffusion of Be into S. S.; failure occurred in the Be
2	Vacuum 7×10^{-5} torr	--	--	129	Fine partition line periodically observed at 300 magnifications

3

Table XIII. Summary of Diffusion Welding Da

Alloy and Condition	Specimen Geometry	Surface Preparation*	Intermediate	Welding Method	Welding Temp, °F	Weldi Press psi
2219-T62 Al to 321 S.S.	Single lap	Wiped with MEK after plating, 5 min before bonding	0.00015-in. Ag plate on both specimens		500	26,000
	Ditto	Rubbed with 320 SiC paper followed with MEK	None		600	21,400
	Ditto, 0.12-in.-thick lapped sheets	Ag plate not cleaned prior to bonding	0.0015-in. Ag plate on both specimens	20-ton press, heated platens	700	15,000 in raised a to defor
	Double lap	Ditto	Ditto	Ditto	Ditto	Ditto
B18S Al to 303 S.S.	Butted rods 1-in. diam x 1 in.	Al: No. 50 grit belt grind 303: No. 240 grit + H_3PO_4 anodic etch	None	Press, sleeve dies, flame heated	750-850	10,000 at 18,000 at
2024 Al to 1020 teel	Single lap 1/2-in. overlap by 1/2 in. wide by 1/4 in. thick	1020 rubbed with alcohol; 2024 cleaned with NaOH and with HNO_3	0.005-in.-thick BAg10 braze alloy	Press	800 optimum	2400 optin

*MEK - methyl ethyl ketone.

Welding Data for Joining Aluminum to Stainless Steel

	Welding Pressure, psi	Welding Time, hr	Welding Atmosphere	Weld Strength, psi	Type of Failure	Reference	Comments
	26,000	3.5		12,800 and 12,900		77	Complete solid-state diffusion; these are best conditions found
	21,400	4.0		1,730 and 450		77	
	15,000 initially, raised at 700°F to deform Al	1/3		17,690 16,410 15,800		77	Deformation limited to 0.004 in.
	Ditto	Ditto		14,500 15,880 14,280		77	Ditto
	10,000 at 850°F 18,000 at 750°F	1/12	Air	31,000-50,000		130	B18S: Al-4Cu-2Ni-0.7Si-1.5Mg
am	2400 optimum	2 optimum	Air	4600 max		131	Less than 3 percent deformation

2

ainless Steel

re	Weld Strength, psi	Type of Failure	Reference	Comments
	12,800 and 12,900		77	Complete solid-state diffusion; these are best conditions found
	1,730 and 450		77	
	17,690 16,410 15,800		77	Deformation limited to 0.004 in.
	14,500 15,880 14,280		77	Ditto
	31,000-50,000		130	B18S: Al-4Cu-2Ni-0.7Si-1.5Mg
	4600 max		131	Less than 3 percent deformation

3

Section VI. OTHER AREAS NEEDING RESEARCH

This report has attempted to provide some understanding of two solid state joining processes, diffusion welding and roll welding, by surveying, summarizing, and evaluating pertinent literature sources dealing with these subjects. In the field of diffusion welding, it is apparent that further research and development is needed in several areas outlined below. Because only a limited amount of effort was devoted to surveying the practice of roll welding, a more thorough evaluation of the state of the art is necessary before an accurate delineation of potentially valuable studies can be presented. Some general comments concerning roll welding can be made, however.

For most joining processes, it appears that a certain amount of development would be required for the fabrication of specific structures. Development of joining procedures, processing parameters, equipment, and hardware and joint designs would be necessary. Secondly, because the majority of solid state joining information has been obtained on a laboratory scale, in terms of specimen size, production rate, and procedures, alterations to meet the needs of users of hardware fabricated by solid state welding would probably also be needed. For example, laboratory parts to be diffusion welded can be cleaned by wire brushing or even ion bombardment and then welded in ultrahigh vacuum. On a larger scale, such practices may be precluded by equipment or time limitations.

Once diffusion or roll welding is recommended as a potential method for fabricating a specific component, its design must incorporate the requirements that are set by the joining technique to be used or evaluated. Based on the particular problems encountered, alterations in the design, welding equipment, procedures, or materials might be warranted in order to achieve the properties needed in service, lower production cost, faster production, or even the ability to fabricate the part at all. These problems all require consideration and possibly research in addition to development. A simple example of this is the cladding of plates, foils, or fuel elements that can be accomplished by press welding, differential thermal expansion welding, gas pressure bonding, and roll welding. Selection of a welding method in this case requires consideration of the factors given above for the particular piece of hardware.

Examples of diffusion welded couples of beryllium to aluminum were not encountered in the literature survey. If an application for a bi-metallic, diffusion welded structure of these materials was found,

development of a joining process would be required. There is no doubt that these materials could be joined by diffusion welding, but such a structure would probably have to meet other criteria also.

Developing nondestructive testing techniques for solid state welded joints is a complex task. Correlating nondestructive test results with reliability and strength demands examination of the specific part under its intended service conditions coupled with destructive tests. The selection and evaluation of a particular test method must be based on the important aspects that were presented in this report.

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13. ABSTRACT

This report presents the results of a state-of-the-art survey of solid state diffusion welding and roll welding of aerospace metals. Particular emphasis is given to beryllium, high-strength aluminum alloys, and Type 321 stainless steel.

The report is divided into four major categories:

1) A general survey of solid-state diffusion welding, including a description of a real surface, factors influencing microscopic plastic flow, diffusion, grain growth, dissimilar metal welding, and use of intermediate materials.

2) A description of solid state welding methods, including roll welding, roll-welding theory, advantages and limitations of welding methods, and joint designs that are suitable for diffusion welding and roll welding.

3) Nondestructive testing techniques for solid state welded joints and their applicability.

4) A survey of similar and dissimilar metal diffusion welding the selected metals.

On the basis of the information obtained, general areas in which future research is desirable are presented.

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